November 1953 Pages 393-480

The Journal
of the
SOCIETY OF DYERS AND COLOURISTS

Volume 69 Number 11

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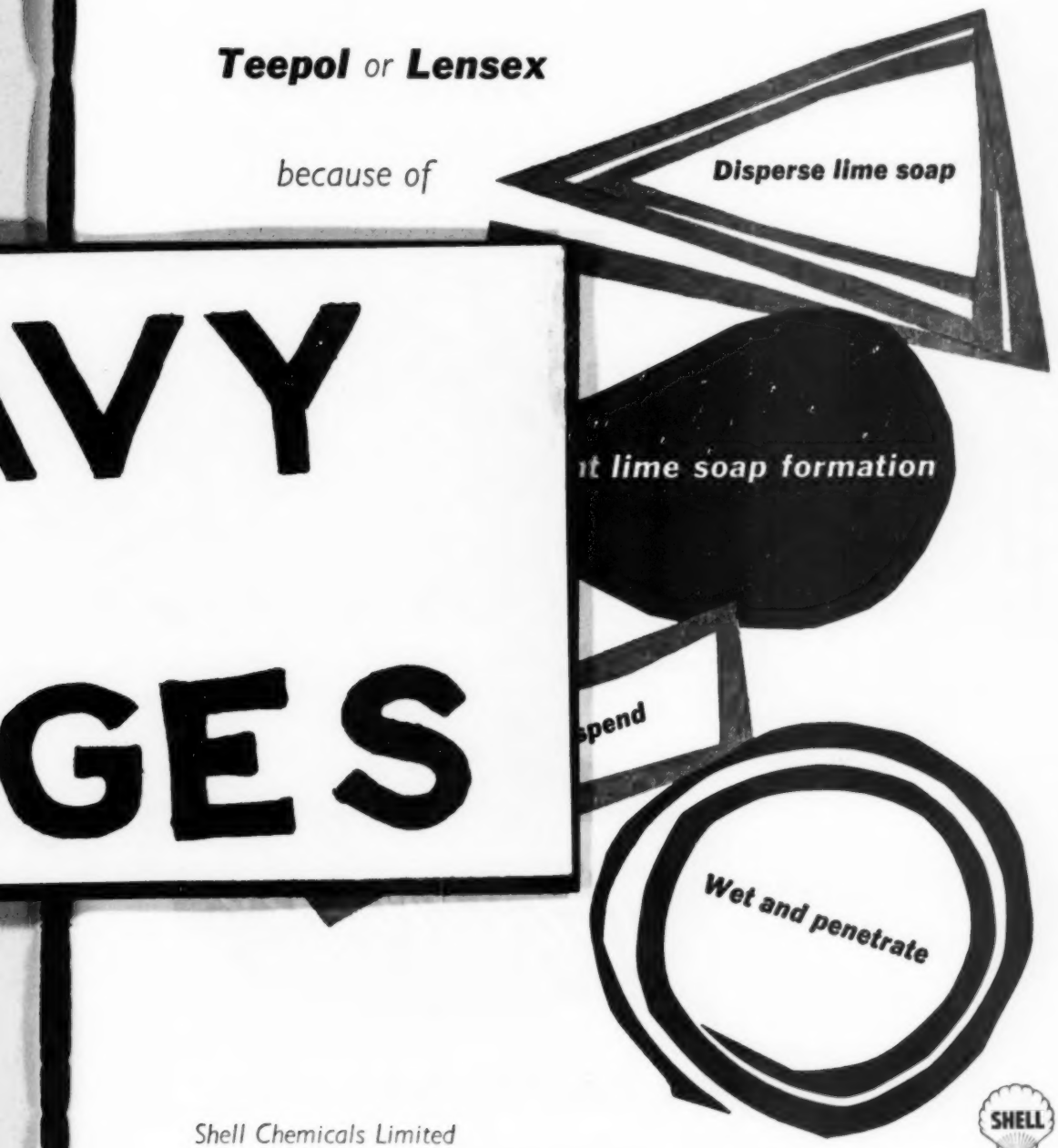

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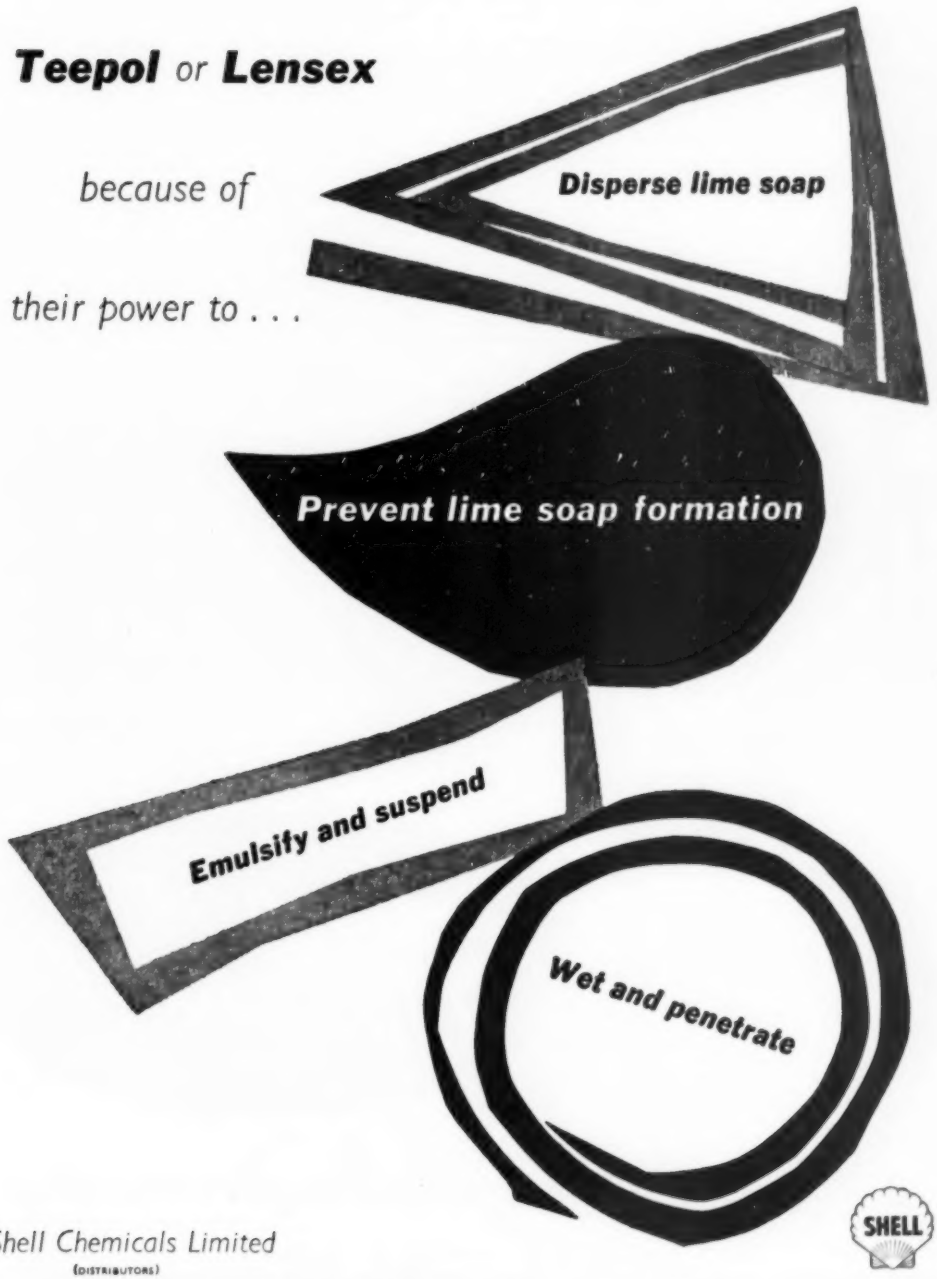

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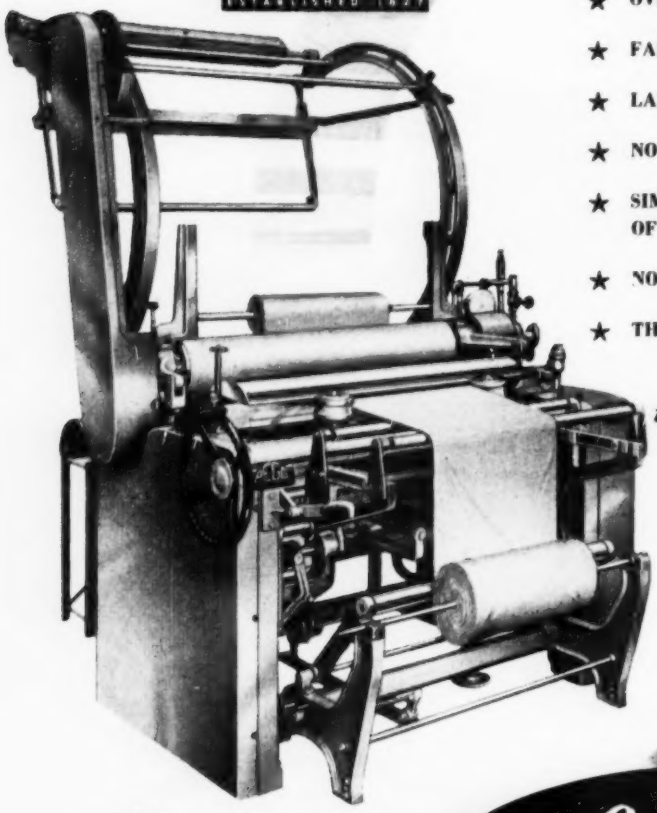

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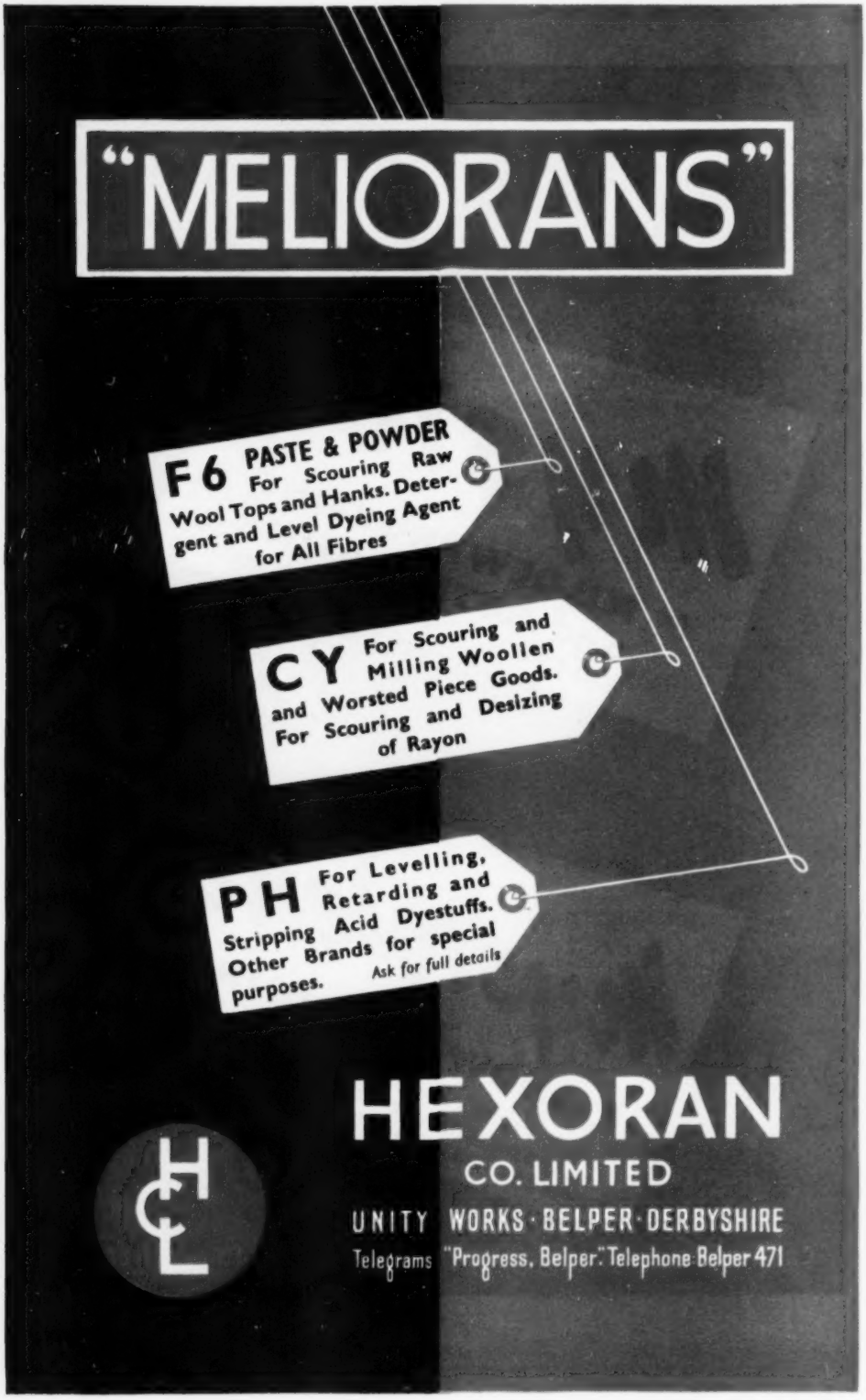
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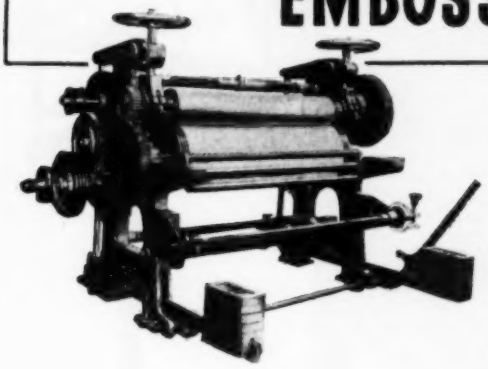

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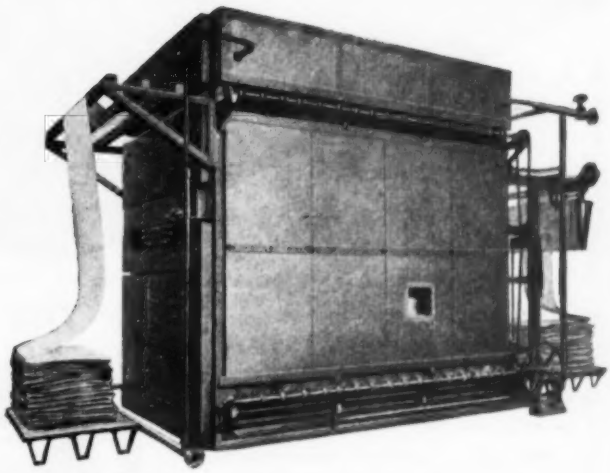

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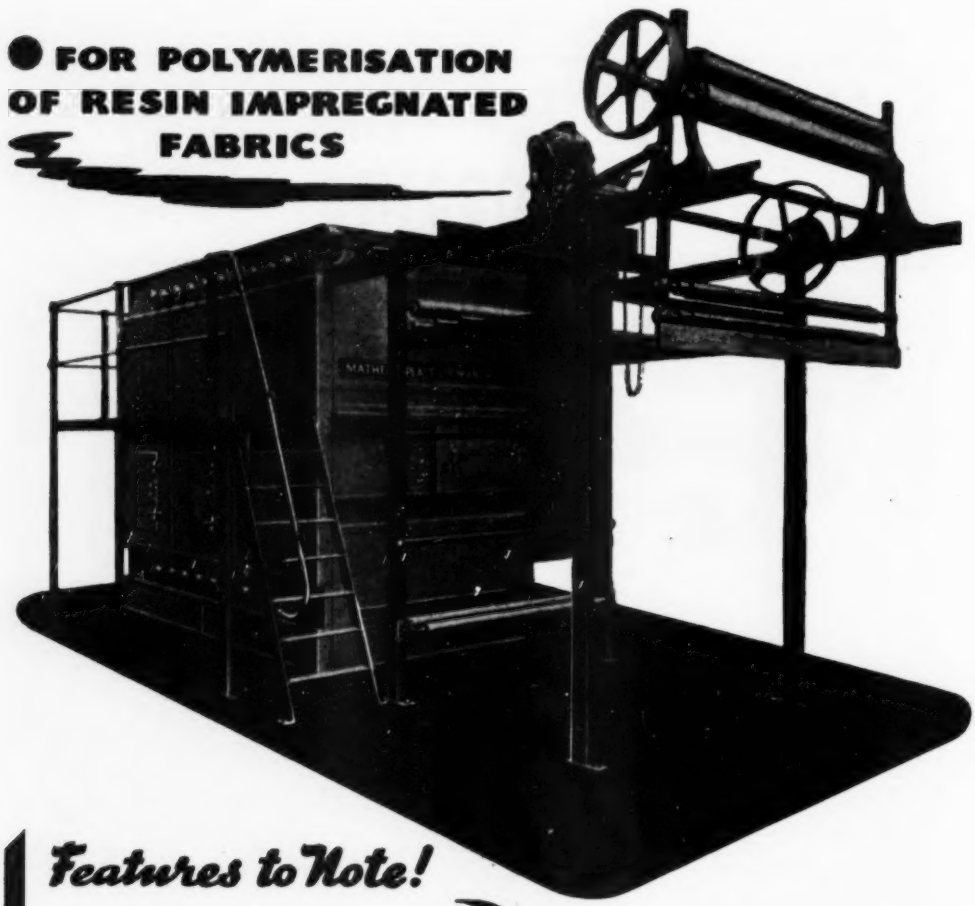

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FORTHCOMING MEETINGS OF THE SOCIETY (see also page xxxv)

SCOTTISH SECTION

All Meetings in St. Enoch Hotel, Glasgow, at 7.0 p.m., unless otherwise stated

1953	
Thursday 10th Dec.	Dr. J. F. Gaunt (Messrs. Patons & Baldwins Ltd.), Scottish Woollen Technical College, Galashiels. *A discussion on the Relative Merits of Different Methods of Applying Chrome Dyes*
Tuesday 15th Dec.	Prof. P. D. Ritchie. *Links between the Textile and Plastics Industries*
1954	
Tuesday 19th Jan.	F. H. Marsh, Esq. (Longclose Engineering Co. Ltd.). *Modern Developments in Dyeing Machinery*
Tuesday 16th Feb.	LADIES' EVENING — Miss J. M. Young, B.A. (I.W.S.). *Fashion History in Wool.* Institution of Engineers and Shipbuilders, Scotland The lecture will be illustrated by a colour film strip and a sound film *Paris, City of Fashion* will be shown also to supplement the lecture.
Tuesday 23rd March	ANNUAL GENERAL MEETING OF THE SECTION. To be followed by three short papers by Sectional members — details to be announced later

MIDLANDS SECTION

Except when stated otherwise, Lecture Meetings commence at 7.0 p.m.

1953	
Wednesday 2nd Dec.	R. W. Speke, Esq., B.Sc. *A Study of Variables in Padding.* Black Boy Hotel, Nottingham
1954	
Wednesday 20th Jan.	J. Payne, Esq., B.Sc. *Silicon Chemicals in Textile Finishing.* College of Technology, Leicester
Wednesday 24th Feb.	Douglas Haigh, Esq., A.T.I. *Some recent researches in the Dyeing of Felt.* Masonic Hall, Nuneaton
Thursday 4th March	R. F. Wilson, Esq. (B.C.C.) (Joint meeting with the Nottingham Textile Society). *Colour.* Gas Theatre, Nottingham
Wednesday 17th March	G. H. Lister, Esq., B.Sc., Ph.D. *The Migration Properties of Acid and Chrome Dyes.* Victoria Station Hotel, Nottingham
Friday 26th March	MIDLANDS SECTION DINNER. King's Head Hotel, Loughborough
Wednesday 7th April	ANNUAL GENERAL MEETING OF SECTION. J. A. Potter, Esq., M.A. *Dyeing and Finishing Synthetic Fibres in the Hosiery Industry.* King's Head Hotel, Loughborough

LONDON SECTION

All Meetings will be held at the Royal Society, Burlington House, Piccadilly, London W.1, at 6.0 p.m. unless otherwise stated

1953	
Thursday 3rd Dec.	Alec B. Hunter (Warner & Sons Ltd.). *Design and Colour*
1954	
Thursday 7th Jan.	Dr. G. T. Douglas (I.C.I. Ltd.). *The Dyeing of Synthetic Fibres*
Friday 5th Feb.	C. P. Tattersfield (Courtaulds Ltd.). *Dyeing and Finishing in relation to Use-Development Work*
Friday 5th March	*The London Lecture.* Subject, Speaker and venue to be given

MANCHESTER SECTION

Unless otherwise stated, meetings take place at the Textile Institute, 10 Blackfriars Street, Manchester 3, and commence at 6.30 p.m

1953	
Friday 18th Dec.	Details to be announced later
1954	
Friday 15th Jan.	S. Blackburn Esq., and M. R. Fox, Esq. (I.C.I. Ltd., Dyestuffs Divn.). *The Behaviour of Leuco Vat Dyes above 100°C.*

WEST RIDING SECTION

Meetings held at the Victoria Hotel, Bradford, at 7.15 p.m. unless otherwise stated

1953	
Thursday 26th Nov.	H. A. Hargreaves, Esq., B.Sc.Tech., A.T.I. (I.C.I.Ltd.). *Small Scale Processing Methods in the Evaluation of New Textile Fibres*
Thursday 10th Dec.	R. F. Wilson, Esq., F.R.S.A. (British Colour Council). *Colour for Increased Production, Safety and Health*
1954	
Thursday 14th Jan.	G. W. Wendon, Esq., B.Sc., Ph.D., A.R.C.S., D.I.C. (English Metal Powder Co. Ltd.). *Preparation, Use and Evaluation of Metallic Pigments.* Metropole Hotel, King Street, Leeds, at 7.15 p.m.
Friday 22nd Jan.	LADIES' EVENING
Thursday 11th Feb.	F. H. Marsh, Esq., B.Sc., F.R.I.C. (Longclose Engineering Co. Ltd.). *Modern Trends in the Manufacture of Dyeing Machinery with Particular Reference to High Temperature Dyeing*
Thursday 25th Feb.	A. R. Smith, Esq., B.A., B.Sc., A.R.I.C. (British Industrial Plastics Ltd.). *The Application of Resins to Textiles*
Thursday 11th March	Prof. J. B. Speakman (The University, Leeds). Lecture. Title to be announced later
Thursday 25th March	LECTURE AND ANNUAL GENERAL MEETING
Tuesday 12th Jan.	S. Kershaw, Esq., F.T.I. (By invitation of the Yorkshire Section of the Textile Institute). *Experiences on a United Nations Assignment.* Midland Hotel, Bradford, at 7.15 p.m.
Monday 8th March	G. Kenneth Seddon, Esq. (By invitation of the Bradford Textile Society). *Interesting Points in Dyeing and Finishing.* Midland Hotel, Bradford, at 7.15 p.m.

NORTHERN IRELAND SECTION

All Meetings with the exceptions of "Ladies' Evening" and "du Pont Story" to be held in Royal Avenue Hotel, Belfast, at 7.30 p.m.

1953	
Friday 4th Dec.	J. V. Summersgill, Esq. (Geigy Co. Ltd.). *Some Aspects of the Chemistry and Textile Uses of Sequestering Agents*
Thursday 10th Dec.	*du Pont Story.* To be held in Thompson's Restaurant, Belfast, at 7.30 p.m. This is a colour/sound film. (Joint Meeting with Textile Institute)
1954	
Friday 8th Jan.	C. P. Tattersfield, Esq. (Courtaulds Ltd.). *Developing New Uses for Rayon.* (Joint meeting with Textile Institute)
Friday 5th Feb.	W. Ford-Kirkpatrick, Esq. *Putting the Colour into Photography*
Friday 5th March	J. Potter, Esq. (Clayton Aniline Co. Ltd.). *Special Finishes for Textiles and Their Evaluation.* (Joint meeting with Foremen Dyers Guild)
Late March or early April	ANNUAL GENERAL MEETING AND DINNER. (*Details later*)

Bradford Education Committee

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NOTICE TO MEMBERS AND SUBSCRIBERS

Readers requiring *general information* regarding the Official Notices, List of Officers of the Society, etc. should consult pages 1–6 of the January 1953 and pages 229–232 of the July 1953 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire (*Telephone* Bradford 25138–9). *Editorial Communications* should be addressed to *The Editor*, at the same address.

Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

LECTURES

The Textile Trade and the Newer Fibres — *F. H. Clayton*

Pressure Dyeing and Bleaching with special reference to Recent Developments — *F. H. Marsh*

The Heat Setting of Terylene Polyester Filament Fabrics in relation to Dyeing and Finishing — *D. N. Marvin*

Studies in the Printing of Terylene Polyester Fibre — *A. G. H. Michie*

The Dyeing of Nylon Yarn in Package Form — *W. S. Willson*

COMMUNICATIONS

The Reaction between Wool and Nickelammonium Hydroxide.

II—The Supercontraction of Wool Fibres in Solutions of Nickelammonium Hydroxide

J. W. Bell and C. S. Whewell

The Dyeing of Acetate Rayon with Disperse Dyes.

I—Aqueous Solubility and the Influence of Dispersing Agents

II—The Relation between Aqueous Solubility and Dyeing Properties — *C. L. Bird*

Absorptiometric Colorimetry in the Textile Laboratory

C. H. Giles and I. S. Shaw

The Effect of Dyeing on the Sorption of Water Vapour by Wool

P. Larose

The Effect on Wool of Boiling in Aqueous Solutions.

I—Water and Solutions of Sodium Sulphate at pH 1·5–9·0

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THE JOURNAL
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Volume 69 Number 11 NOVEMBER 1953 *Issued Monthly*

Message from the President

At the recent Symposium on Textile Printing held at St. Annes-on-Sea it was my privilege to announce that the Council of this Society had decided to institute two professional qualifications— the Associateship and the Fellowship of the Society of Dyers and Colourists— with two principal objects in mind.

For some years the Council, stimulated by the views of many ordinary members of the Society, has felt that there is a need to encourage an improved type of educational course for young people wishing to enter the colour industries. Systems of instruction at present available, whilst in many cases excellent, do not always cover the training which is desirable for some parts of the industries served by the Society. In addition, the Council was aware of a strong feeling amongst the membership that existing academic and other qualifications were not a very reliable guide to prospective employers as to the suitability of persons holding these qualifications for employment in particular fields of the colour industries.

These two principal objects had as a background an increasing national consciousness of the importance of improvement in number and quality of technologists. The Council felt that the establishment of new qualifications sponsored by the Society would fill the gap in present qualifications, and also that the very existence of the Society's qualifications would encourage provision in educational institutions of technological courses more suited to the needs of our industries.

In setting its hand to such a project the Council realises that the Society is undertaking an exacting and responsible task, but is of the opinion that it is a task which is really a duty laid upon it by virtue of its principal object, viz. the advancement of tinctorial technology. We are therefore embarking upon the venture in the full confidence that what we propose to do is right, since it will fulfil a real need, but conscious of the necessity for the active support of every member of the Society, and of the goodwill of other societies. I am glad to be able to say that in a large measure other societies with whom we have taken counsel have assured us of their general approval of the steps which we are taking.

There are obviously a large number of matters connected with the administration of the scheme which the Society's Diploma Committee is busy working out in detail, but the hub and kernel of the whole scheme is the standard of the proposed qualifications. This must be sufficiently high to render the new Associateship and Fellowship acceptable to all concerned as worth while and in particular to employers as a true guide to the suitability of a candidate for employment in a particular field of the colour industries.

The regulations for the award of Associateships and Fellowships have been approved by Council and are published in this issue of the *Journal* in order that all interested members and others may have as early information as possible of the standards which the Society proposes to establish and maintain. It is perhaps noteworthy that one of the essential features of the regulations is that neither Associateship nor Fellowship will be conferred on any candidate without evidence of a period of practical experience in the field of colour technology which the candidate intends to practise.

I commend to all members of this Society the earnest study of the regulations now placed before you.

F. L. Goodall

THE SOCIETY OF DYERS AND COLOURISTS

Regulations governing Admission of Registered Students and the Election of Associates and Fellows of the Society of Dyers and Colourists

Rules and Syllabuses for the Associateship Examinations

The Society of Dyers and Colourists confers diplomas in tinctorial technology on suitably qualified members. These distinctions are in two grades—the Associateship and the Fellowship—and confer the right to use the title Associate of the Society of Dyers and Colourists (A.S.D.C.) or Fellow of the Society of Dyers and Colourists (F.S.D.C.). To obtain the Associateship candidates are required to pass the prescribed examinations and to furnish evidence of satisfactory general education and of training and experience in at least one branch of the manufacture or application of colouring matters. The Fellowship is conferred on senior members who have attained high standing in the knowledge and practice of tinctorial technology. Students who intend to take the Society's examination are strongly recommended to become Registered Students, so that their studies and preparation may be effectively directed.

Regulations

Registered Students

A Registered Student shall—

(*a*) Be a member of the Society

(*b*) Have reached the ordinary standard of the General Certificate of Education in English language, mathematics, and two other subjects, have passed equivalent examinations, or otherwise have satisfied the Council that he has attained an equivalent standard

(*c*) Be following an approved course of instruction and produce evidence annually that attendance and studies are satisfactory

(*d*) Make application on the prescribed form and accompany it with a fee of 5s. 0d.

Associateship

An Associate shall—

(*a*) Be a member of the Society

(*b*) Have passed the prescribed examination

(*c*) Have satisfied the Council that in personal character and professional attainments he is worthy of the distinction

(*d*) Have paid the prescribed fees.

Fellowship

A Fellow shall—

(*a*) Be a member of the Society and at least thirty years of age

(*b*) Have (i) been awarded the Associateship and subsequently have had at least four years' experience in an approved technical occupation in one or more branches of tinctorial technology,

or (ii) produced evidence of at least ten years' approved training and practice in tinctorial technology

(*c*) Have (i) presented a satisfactory dissertation on an approved subject,

or (ii) submitted evidence of having contributed effectively to the advancement of tinctorial technology,

or (iii) submitted satisfactory evidence of having attained distinction in the practice of tinctorial technology or of a branch of industry related thereto

(*d*) Have satisfied the Council that in personal character and professional attainments he is worthy of the distinction.

(*e*) Have paid the prescribed fees.

Rules and Syllabuses for the Associateship Examinations

Rules

A candidate shall—

(1) Apply on the prescribed form, on which he shall—

(*a*) Provide evidence of the attainment of a satisfactory standard of general education

(*b*) Be supported in his application by three Fellows

(*c*) Indicate the special branch of tinctorial technology in which he desires to be examined and whether he desires to take the examination in one or two stages.

(2) Pay the prescribed fees.

(3) Before taking papers C, D, and E, as described below, produce evidence of having satisfactorily followed for five years after attaining the age of seventeen years an approved occupation in one or more branches of tinctorial technology. From this period of five years a reduction of not more than two years may be allowed to a candidate who has satisfactorily completed an approved course of instruction, part or full time.

(4) Provide details of his training and experience.

Examination

The examination consists of five written papers and an interview at which the general personality, resourcefulness, and sense of responsibility of the candidate will be assessed. When it may be unreasonable to ask the candidate to attend for interview, the Council will take such other means as it may deem necessary to assess the suitability of the candidate.

The candidate may take all five papers together, or alternatively may first take Papers A and B

together, and subsequently take Papers C, D, and E together to complete the examination.

The scope of each written paper is—

PAPER A— General chemistry and properties of dyes, pigments, auxiliary products. General chemistry and properties of organic high polymers.

PAPER B— Theories of colour. Relation of colour to constitution. Colour assessment and colour fastness.

PAPER C— Properties of the materials and finished products and fundamental mechanisms of the industrial processes in a specific branch of tinctorial technology.

PAPER D— Industrial procedures and plant in the branch chosen for Paper C.

PAPER E— Analytical and testing techniques or the organisation of production and economics in the branch chosen for Paper C.

The branches of tinctorial technology referred to under C, D, and E shall be—

1. Textiles
2. Leather
3. Skins and furs
4. Paper
5. Paints, lacquers, printing inks, etc.
6. Coated fabrics, leathercloths, linoleum, etc.
7. Rubber and plastics
8. Substrata not dealt with under one of the above
9. Production of dyes, lakes, and pigments.

APPENDIX

The scope of technical knowledge in each of the branches of tinctorial technology is given below.

1. TEXTILES— Chemical and physical properties of the textile fibres, manufacture of yarns and fabrics; preparative, cleansing, and finishing processes for industrial textile products— all considered in relation to the processes of coloration or to the properties of the coloured materials. Theories of dyeing, assessment of dyeing properties. Coloration of all commonly encountered textile products. Methods for assessing quality of coloured textile products. The papers set will demand (*a*) adequate general knowledge of the properties of textile materials and their processing, and (*b*) detailed knowledge of a particular branch of textile coloration.

2. LEATHER— Morphology and chemistry of skin, with special reference to cross-linking, denaturation, and other processes for increasing chemical stability and improving mechanical properties. Principles of leather coloration. Varieties of skins used, industrial processes preparatory to tanning, tanning operations, finishing operations— all with references to coloration processes and properties of coloured leather. Detailed knowledge of the varieties of colouring matters applied to leather; industrial methods of application to different kinds of leather and for different purposes, including the production of multicoloured effects. Methods used in the analytical control of the operations and in assessing quality of the finished products.

3. SKINS AND FURS— Morphology of skin and hair, chemistry of skin and hair proteins with special reference to cross-linking, denaturation, and other processes for increasing chemical stability and improving mechanical properties. Principles of coloration of skins and furs. Varieties of skins and furs, methods for their preservation, dressing, and preparation prior to dyeing; selection of dyes and industrial methods of application, treated in full detail; treatment after dyeing. Assessment of quality of the finished products.

4. PAPER— Materials and processes used in paper making, coating, and staining— all with reference to the coloration of paper and the properties of the coloured material. The different classes of colouring matters used and their application. Assessment of quality of the finished product.

5. PAINTS, LACQUERS, PRINTING INKS, ETC.— Chemical and physical properties of vehicles and other raw materials. Detailed knowledge of the varieties of colouring matters used and of their application. Theory and practice of the manufacture of paints, etc. Assessment of quality of the finished product.

6. COATED FABRICS, LEATHERCLOTHS, LINOLEUM, ETC.— Chemical and physical properties of bases and coating materials. Detailed knowledge of the varieties of colouring matters and of their application to different bases. Theory and practice of preparation of base and coating compositions. Coating operations. Assessment of quality of the finished product.

7. RUBBER AND PLASTICS— Chemical and physical properties of rubber and plastics. Detailed knowledge of the varieties of colouring matters and their application. Theory and practice of compounding, vulcanisation, and curing, especially with reference to coloration. Assessment of quality of the finished product.

8. SUBSTRATA NOT DEALT WITH UNDER 1–7— Suitable papers to be prepared as necessary by the Examiners for any candidate engaged in the colouring of substrata not dealt with under 1–7, e.g. in wallpaper manufacture.

9. PRODUCTION OF DYES, LAKES, AND PIGMENTS— Production and properties of natural and synthetic organic colouring matters, and natural and synthetic inorganic pigments. Standardisation and testing of the finished product.

The candidate will be expected to have a knowledge of the different forms in which dyes and pigments are produced for various purposes.

The questions will be set so that equal opportunity will be given to the candidate specialising in dyes and to the one specialising in lake or inorganic pigment manufacture.

Proceedings of the Society

The Uses of Microscopy in Textile Dyeing and Finishing

G. G. TAYLOR and J. C. BROWN

Meetings of the West Riding Section held at the Victoria Hotel, Bradford, on 14th February 1952, Mr. C. O. Clark in the chair; of the Midlands Section held jointly with the British Association of Chemists at the Midland Hotel, Derby, on 27th February 1952, Mr. A. P. Kershaw in the chair; of the Bradford Junior Branch held at the Bradford Technical College on 14th October 1952, Mr. E. E. Sunderland in the chair; of the Huddersfield Section held at Field's Café, Huddersfield, on 21st October 1952, Mr. E. Rolfe in the chair; of the Northern Ireland Section held in the Queen's Hotel, Belfast, on 12th November 1952, Mr. D. A. Derrett-Smith in the chair; of the London Section held in the rooms of the Royal Society on 9th January 1953, Mr. G. F. Wood in the chair; of the Scottish Section held at the St. Enoch Hotel, Glasgow, on 13th January 1953, Mr. R. D. Alexander in the chair; and of the Leeds Junior Branch held at Leeds University on 17th February 1953, Dr. C. B. Stevens in the chair

The object of this paper is to review the application of microscopy to the study of textile fibres. A brief exposition is given of the best methods of mounting and examining various fibres, and the application of the techniques of polarised light, staining, and microtomy is discussed. Recent advances in the histology of protein fibres due to both visual and electron microscopy are described, and the more useful microscopic tests for protein fibre damage are covered. The structure of the natural cellulosic fibres cotton and linen is discussed, and the microscopical detection of chemical modifications to cellulose is described. An outline is given of some staining experiments which have a bearing on the structure of regenerated cellulosic fibres.

MOUNTING TEXTILE FIBRES FOR MICROSCOPICAL EXAMINATION

Before examination, fibres must be mounted in a suitable medium. For normal routine examination water is generally used, but the use of other liquids with carefully chosen refractive indices is often advantageous. If, for example, we mount filaments of viscose rayon (refractive index n = approx. 1·53) in water (n = 1·33), the main feature of the image is the viscose rayon–water interface because of the comparatively large change in refractive index involved. The characteristic striated appearance resulting from the fibres having an irregular cross-sectional shape is seen, and it is difficult to study any internal structure of the fibre.

If, instead of mounting the fibre in a liquid of comparatively remote refractive index, we chose one almost identical with the fibre in refractive index, the fibre–mountant system becomes optically almost homogeneous, and the fibre is rendered invisible. It is then said to be *optically dissolved*. Any internal structure of different refractive index is now clearly seen, and can be studied. The fibres shown in Fig. 10 are dyed delustred 4·5-denier viscose rayon mounted in ethyl salicylate (n = 1·527). The fibre–ethyl salicylate interface is visible only by virtue of the colour of the fibre, but the particles of titanium dioxide pigment used for delustring (n = approx. 2·6) are immediately obvious. Particle size determinations are thus facilitated.

The list of suitable "optical solvents" in Table I will be found useful.

TABLE I

Fibre	Optical Solvent	Refractive Index
Normal cellulose acetate ...	Pure glycerol (sp. gr. 1·26)	1·473
Regenerated cellulose fibres	Ethyl salicylate	1·527
Animal protein fibres ...	Nitrobenzene	1·553
Silk	Bromobenzene	1·559

Animal protein fibres have an imbricated scale structure [1, 2] which varies in pattern, even between individual varieties of the same fibre. This scale structure is useful for purposes of identification, but is difficult to study with normal mounting techniques. In 1936 a method was perfected by Reumuth [3] of half embedding fibres, so that one half of the fibre was in contact with air and the other half was embedded in a suitable medium. In 1938 Herzog [4] simplified the technique considerably by using ordinary Sellotape (Adhesive Tapes Ltd.), the sticky gum of which has a refractive index similar to that of wool. Fig. 11 shows a medium variety of merino wool mounted by this technique. The fibres are simply placed on a microscope slide and attached to it with Sellotape. A little practice soon indicates how much pressure to apply in order to half-embed the fibres. Examination is carried out directly without the use of a cover-glass, and the surface shown in Fig. 11 is the bottom one of the fibre in contact with air, the top surface having been optically dissolved by intimate contact with the Sellotape. This method of examination has two distinct advantages—

(1) The surface of the scales under examination is in contact with air, and the large change in refractive index at this interface makes every detail visible.

(2) The top surface is invisible, and hence cannot interfere with the image of the bottom surface.

STAINING

To emphasise further some particular feature of a fibre it may be possible to stain the fibre in a useful manner. The type of stain used depends on the available reactive groups in the fibre, but is usually restricted to dyes of the direct, acid, and basic classes. The choice of a stain is also dependent on which structural or other features it is desired to study. For instance, the cotton fibres in Fig. 12

FIG. 1—Linen Fibres viewed between Crossed Polaroids

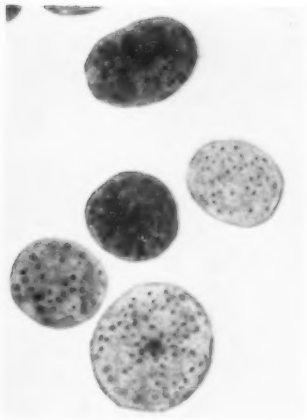


FIG. 2—Stained Mohair Cross-sections

FIG. 3—Pauly Test on Blend of Chlorinated and Unchlorinated Wood

FIG. 4—Methyl Orange Test for Residual Acid in Wood

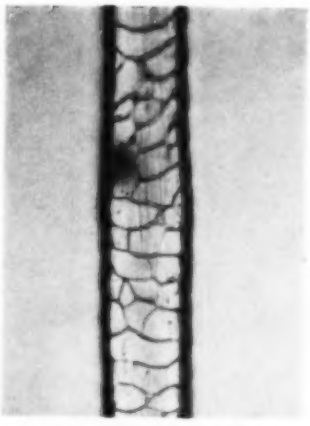


FIG. 5—Normal Wool stained with Victoria Blue B

FIG. 6—Wet-chlorinated Wool stained with Victoria Blue B

FIG. 7—Aftertreated Dry-chlorinated Wool stained with Victoria Blue B

FIG. 8—Cotton viewed under Circularly Polarised Light

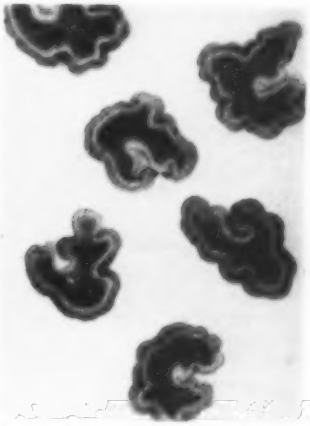


FIG. 9—Skin Formation in Viscose Rayon

FIG. 10—Dyed Delustred Viscose Rayon

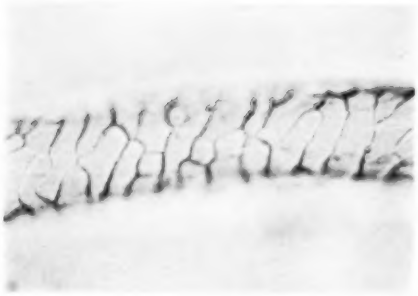


FIG. 11—Merino Wool mounted in Sellotape

FIG. 12—Protoplasmic Residue in Cotton

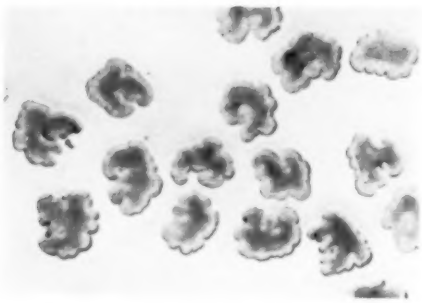
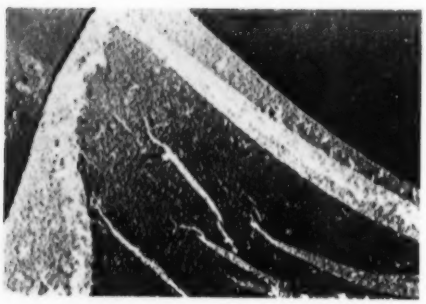


FIG. 13—Cross-sections of Viscose Rayon



[by courtesy of Prof. N. Gralén

FIG. 14—Electron Micrograph of Epicuticle of Wool

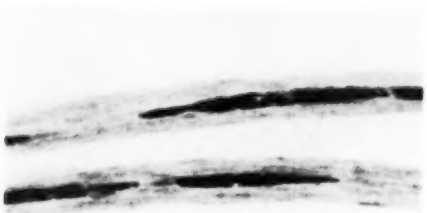


FIG. 15—Alpaca in Nitrobenzene

FIG. 16—Opossum Fibre in Nitrobenzene

FIG. 17—Mechanically Damaged Wood stained with Kiton Red G

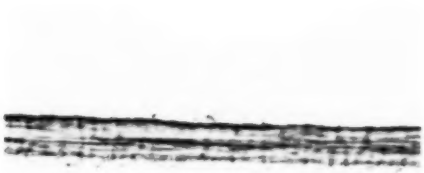


FIG. 18—Allworden Test on Wood

FIG. 19—Krais-Viertel Test on Wool

FIG. 20—Extrusion Test on Normal Cotton

FIG. 21—Congo Red Test on Mechanically Damaged Cotton

FIG. 22.—Congo Red Test on Mildewed Cotton

FIG. 23.—Fibrillated Linen Fibre viewed under Polarised Light

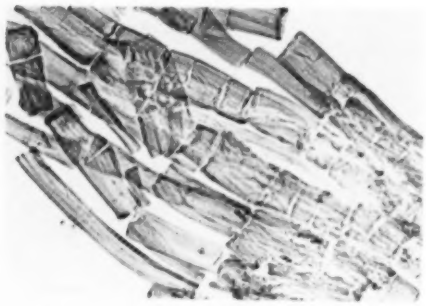


FIG. 24.—Chemically Damaged Linen Fibre

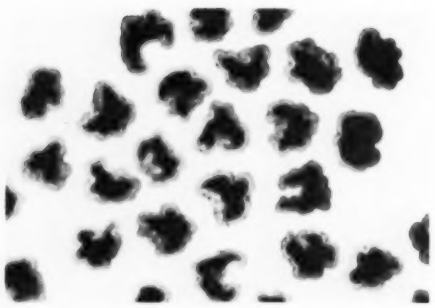


FIG. 25.—Cross-sections of Viscose Rayon stained with Chlorantine Fast Green 5BLL

FIG. 26.—Stained Skin of Viscose Rayon Cross-sections

have been stained with Acid Black HA, which has been strongly absorbed by the protoplasmic residue remaining in the contracted lumen of the fibre. This residue may occasionally be troublesome to the dyer who wishes to reserve cotton fibres while dyeing with wool dyes normally applied from an acid bath.

Staining is a highly developed and accurate branch of biological microscopy, but its application to textile microscopy seems to have been somewhat neglected.

USE OF POLARISED LIGHT

Although microscopes are sold with expensive attachments for polarised light using Nicol prisms, much useful work can be carried out with self-made attachments of Polaroid sheet costing a few shillings. Polarised light is of some advantage in difficult cases of fibre identification. Some of the finest qualities of linen, for instance, yield extremely regular, well ordered fibres which might be mistaken for mercerised cotton, or even cuprammonium filaments, by the inexperienced operator. When the fibres are viewed by polarised light, however, the pronounced nodes or dislocations which occur at irregular intervals along the length of the fibre stand out clearly (Fig. 1) and are characteristic of linen. The colours produced in a fibre viewed between crossed polaroids are largely determined by its birefringence, and as this varies considerably from fibre to fibre the possibility of a useful analytical method suggests itself. A procedure based on this principle has recently been elaborated by American workers [5], which should help to cope with the rapidly growing identification problem in the field of synthetic fibres and which possesses the virtue of simplicity. Mention will be made later of an application of circularly polarised light.

PHASE-CONTRAST MICROSCOPY

The usefulness of visible-light microscopy has been extended recently by the development of the method of phase contrast, whereby small differences in optical path length of different parts of an object, leading to phase differences in the final image, are rendered visible as intensity differences[6]. It is interesting to note that the epicuticle of wool, which required the electron microscope for its discovery, can be observed in the visible microscope by means of the phase-contrast method [7]. The application of this method to textile fibre research has been reviewed by Reumuth [8].

MICROTOMY

Another technique which is extremely useful in textile microscopy is the cutting of fibre cross-sections. Several simple and rapid methods have been suggested for doing this, but they all give inferior results to the classical methods developed by histologists for dealing with biological materials. Schwarz [9] used a metal plate with a very small hole in it, through which the fibres are pulled by means of a fine wire. The protruding ends are then cut off on either side of the plate with a razor blade, when the fibres are ready for examination. This method is really suitable only for undyed fibres, and it is not possible to see any internal structural features, since only the shape of the ends of the fibres is examined. A simple form of hand microtome has been developed by Hardy [10] which enables a skilled operator to cut fairly thin sections without the use of embedding media. This technique has the defect that the fibres are liable to be deformed by pressure. Fig. 13 illustrates some cross-sections of 4·5-denier viscose rayon fibres prepared by the satisfactory, though laborious, method of embedding in paraffin wax and sectioning on a mechanical microtome. The stages in preparation involve dehydration with alcohol, "fixing" with xylene or chloroform, and embedding in paraffin wax in a suitable mould. The wax block is eventually sectioned on a microtome, which should preferably be of the massive sledge type, in which, if necessary, the knife can be set at an angle to the direction of cut and the slice made along the entire length of the blade.

Recently more flexible embedding media, which are easier to work with, have been devised and used by many workers. Moore *et al.*[11] have made use of methyl methacrylate (Perspex (ICI) monomer) with a suitable proportion of plasticiser, which can be polymerised with the fibres *in situ* to any degree of hardness depending on the proportion of plasticiser used. Rapid polymerisation is brought about by addition of 0·5% benzoyl peroxide followed by heating for 15 min. at 58°C.

Application to Protein Fibres

It is many years ago that textile microscopists first recognised the existence of two different types of protein fibre cells—the spindle-shaped cortical cells, which make up the bulk of the fibre, and the scale-like cuticle cells, which form an outer protecting sheath. It is now known with certainty that both these cell types are structurally complicated, and can be divided into different components. In addition, the biological aspects of their formation have been extensively studied [12].

The cortical cells of wool were shown by Hock *et al.* in 1940 [13,14] to have a fibrillate structure, and this was confirmed in 1949 by means of the electron microscope. These workers also differentiated the nucleus of the cortical cell by staining with Orange II and mounting individual cortical cells. Fig. 2 shows some cross-sections of winter mohair fibres which have been stained for a few minutes with a cold acidified solution of Kiton Red G. Apparently there is a very rapid initial absorption of colour by the cortical cell nuclei, since they are very clearly stained.

ELECTRON MICROSCOPY

The electron microscope has also been used to investigate the finer structure of the cuticle cell [2,15] Gralén and his co-workers at the Swedish Institute for Textile Research in Gothenburg have described the cuticle as being covered with a fine membrane, which they have called the *exocuticle* and about which relatively little is known [15–17]. There is also a final outer layer, called the *epicuticle*, which is extremely thin (about 100 A.) and very resistant chemically [15]. It seems not unlikely that many of

the properties of the wool fibre, such as rate of wetting, rate of uptake of dye, etc., will eventually be explained partly in terms of this epicuticle.

Fig. 14 is an electron micrograph of an epicuticle covering at a magnification of approx. 20,000, produced in Gothenburg by digesting the fibre with sodium sulphide. The extreme thinness of the epicuticle is strikingly illustrated where the membrane is folded over on itself.

Many chemically different treatments give an epicuticle of similar appearance and thickness, proving it, beyond doubt, to be a component of the wool fibre, and some show adhering material believed to be the exocuticle. Apart from animal hairs, the epicuticle seems also to be a feature of other keratinous substances such as feathers, and it is interesting to note that recently similar membranes have been isolated from human skin and from human fingernail parings [18]. Another structural component of wool, whose existence has been claimed by many workers in the past and more recently by Alexander and Earland [19], is thought to exist between the cuticle and the cortical cells, and can be referred to as the *subcutis* or *subcuticle* [20,21].

IDENTIFICATION OF PROTEIN FIBRES

The individual protein fibres commonly met in textile work, such as wool, cashmere, mohair, alpaca, etc., are fairly easily recognised by direct observation under the microscope, although it should always be borne in mind that within one fibre there are often many widely different varieties. It may be necessary when in doubt to examine the scale structure by the Sellotape method, and always the most certain method is direct comparison with a large and reliable collection of authentic samples. Fig. 15 shows a few fibres of alpaca mounted in nitrobenzene, and illustrates the usefulness of the natural pigmentation as a means of characterising this fibre. Another variable and useful feature of natural protein fibres is the medulla cell structure. Sometimes the internal pigment of these cells is absent, and the resultant cavities have become filled with air. The size, the shape, and the distribution of these cavities are sometimes characteristic, the large square-shaped ones of the rabbit family, for instance, being well known. Fig. 16 illustrates an opossum fibre mounted in nitrobenzene.

TESTS FOR FIBRE DAMAGE

We can now turn our attention to the practical application of this knowledge, and see how it enables us to approach intelligently a bewildering number of different tests for fibre damage and modifications which have steadily accumulated in the literature. The cuticle cell being more resistant to chemical penetration than the mass of the fibre, any damage, whether mechanical or chemical, will tend to increase the initial rate of absorption of a reagent. The reagent may be a dye or may be a substance which can form a coloured complex with one of the many reactive side-chains of the keratin molecule. Fig. 17 is a photomicrograph of mechanically damaged wool stained with a 0·1% solution of Kiton Red G 275% (*C.I.* No. 31) in the cold for 10 min. with agitation [22,23]. The staining along the fibre body is restricted to those regions where the cuticle cells have been ruptured or displaced by damage. Although this stain has been suggested to detect many different types of damage, it is almost completely non-specific, and will give a positive reaction to many different treatments. Semple [24] and others have used this stain in conjunction with a photoelectric method of estimating fibre damage, in which the type and the amount of staining are determined optically, after mounting in cedar wood oil. There are many more stains of this type which have been suggested from time to time, e.g. Benzopurpurin 10B introduced by Sieber in 1928 [25], but they do not differ sufficiently to warrant individual mention.

Bacterial attack and mildew action on wool share with mechanical damage the property of being easily recognised, since they cause intense intercellular disintegration, which often causes the fibre to break down completely, producing "brush-like" ends [26].

Tests involving Tyrosine Side-chain Reactions

The Pauly test, introduced by Pauly and Binz in 1904 [27], uses diazotised sulphanilic acid, which can couple with the amino acid tyrosine to give an azo dye of high colour intensity, but is similar to stains of the acid or basic dye class in that development of colour on the fibres is proportional to the damage suffered by the cuticle cells. Staining should always, therefore, be carried out for a standard period of time. Fig. 3 shows a selection of wool fibres stained with the Pauly reagent which were taken from a sample of half-hose consisting of a blend of chlorinated and unchlorinated wool. This had been severely damaged by being stripped and redyed several times. The coloured complex formed is stable to alkaline hydrolysis, and it was suggested by Rimington in 1930 [28] that this test could be made more quantitative by hydrolysing the stained wool fibres and estimating the resultant coloured solution colorimetrically, the chemically identical dye New Acid Brown S being used as a standard. The various workers who used this stain recognised also that diazotised sulphanilic acid can couple with other amino acids [29]: for instance, a somewhat yellower shade is obtained with histidine (α-amino-β-4-glyoxalinylpropionic acid), but apparently this is sufficiently close to that produced by tyrosine not to interfere seriously. The mechanisms of this and other similar diazo reagents have recently been re-investigated by Glynn [30].

The Allwörden Reaction

The tests just described can only be said to have a limited application, since they are extremely non-specific. While they are of considerable value in the semi-quantitative estimation of fibre damage where the damaging agent is known, they are often of little value where the nature of the damage itself is unknown. Most of the tests for fibre damage which claim to recognise different chemical actions on the fibre are swelling rather than staining tests. For instance, it was first pointed out by Allwörden in 1916 that aqueous solutions of halogens produce

characteristic sac-like swellings on the wool fibre, at a rate which depends on the previous history of the sample [31].

Although Allwörden used chlorine water, subsequent workers favoured the slightly slower bromine water. In Fig. 18 it can be seen that these swellings are composed of an aqueous solution held by an outer flexible membrane, as has been demonstrated by puncturing the sacs with a micro-needle, when they are observed to collapse.

The membrane itself, which can hardly be anything other than the epicuticle observed by Gralén under the electron microscope [15], is almost too thin to be seen, and is rendered visible only because the liquid inside the sac is of a different refractive index from that outside [32]. Considerable confusion has existed as to the value and interpretation of this test in connection with alkaline damage, and it has recently been claimed by Meeuse *et al.* of the Vezelinstituut in Holland that bubbles are always formed in the presence of intact scales and that it is the time taken for these to appear which varies according to the previous history of the sample [33]. These workers have recently published an excellent monograph [33], containing an almost complete summary of the literature up to 1948 and reviewing the whole field of damage to wool fibres, in which they suggest that the following interpretation of the Allwörden test is reliable. Wool damaged by acids reacts more rapidly, while wool damaged by alkali, heat, or light reacts more slowly, than a corresponding sample of normal wool, although care must be exercised in the presence of chromium, since this element seems to affect the reaction time in some unknown manner.

The Krais–Viertel Reagent

Fig. 19 illustrates the characteristic bulbous swellings produced on the wool fibre by the Krais–Viertel reagent [34–37], which consists of a solution of caustic potash in aqueous ammonia. As in the case of the Allwörden test, the time taken for these swellings to appear is claimed by Meeuse *et al.* [33] to depend on the previous history of the sample. Wool damaged by acids has a shorter reaction time, and wool damaged by alkali, heat, or light a longer reaction time. Several factors affect the results, such as the fibre diameter, the temperature, and the age of the reagent, but, provided that these are under control, the difference between the reaction time of the damaged sample and that of the undamaged wool is proportional to the degree of damage.

Methyl Orange Test for Residual Acid

A useful test for residual acid in fibres is the Methyl Orange test which uses a 0·1% solution of this indicator. The free acid of Methyl Orange is insoluble at this dilution, and is precipitated as fine needles (Fig. 4), which tend to grow round the fibre when sufficient acid diffuses out to bring the pH of the solution below about 4·0 [33]. Calcium salts interfere, but they give characteristic plate-like crystals which cannot be confused with those of the free acid of Methyl Orange.

Staining with Basic Dyes

Chlorination anti-shrink treatments on wool [38] can be detected by use of a basic dye such as Methylene Blue or Victoria Blue B [39]. Fig. 5 shows a normal wool fibre which has been stained with a 0·002% aqueous solution of Victoria Blue B. The fibre is practically unstained with the exception of the very edges of the scales, which presumably have suffered considerable mechanical damage even during normal processing. The effect on the fibre of, say, an aqueous hypochlorite solution is still not completely understood [40], but it appears to react unevenly with the cuticle cells of the wool fibre, since on staining wet-chlorinated wool fibres with Victoria Blue B (Fig. 6) a patchy effect develops which is very characteristic. The staining has spread inwards from the scale edge a considerable distance, the amount of staining being proportional to the severity of the treatment.

If wool is examined under the microscope immediately after chlorination, the appearance is very similar to that of untreated wool. Subsequent chemical treatment or the action of a few domestic washes produces a profound effect. Normal wool fibres show slight modification, but chlorinated wool fibres now swell easily in aqueous solution and most of the scales have been removed. Fig. 7 illustrates a fibre of Woolindras shrink-resist wool which has been given a treatment simulating several severe domestic washes and then stained with Victoria Blue B. The scales are loosened to the point where they are ready to part from the fibre body, and are very heavily stained blue.

Application to Cellulosic Fibres

Our knowledge of natural cellulosic fibres would be very incomplete without the contribution made by microscopists. Cotton, for instance, has been extensively studied [41], and we now possess a fairly complete picture of its internal structure, growth, development, and maturation, while almost all known varieties and abnormalities have been studied microscopically. One of the structural features which has been studied is the spiral fibrillar structure of the cellulose layers. For some reason, as yet unknown, there are places along the fibre where the direction of rotation of these spirals changes, and this has recently been extensively restudied by Wakeman and Spicer [42] at the Textile Research Institute at Princeton, N.J.

These workers have made use of an elegant technique, previously illustrated by Hock [43], whereby the fibre is illuminated by elliptically polarised light. The fibre shown in Fig. 8 was illuminated by elliptically polarised light produced by means of two crossed sheets of Polaroid and a plate of good-quality mica split down to a suitable thickness [44]. The thickness of the mica plate determines the colours produced, since only for a certain wavelength of light and a certain angle of inclination of the optical axis of the mica to the plane of polarisation of the first Polaroid sheet will truly circularly polarised light be produced, and the intensity at this wavelength is least affected by the second or analysing Polaroid sheet. Other wavelengths give elliptically polarised light, and the intensity of

these is considerably reduced by the second Polaroid sheet. This produces the approximately monochromatic character of the background. It may be noted that the colours on either side of the point of reversal are approximately complementary. These reversals occur randomly, about a hundred to the inch, along the fibre, and Wakeman and Spicer were interested in finding a correlation between their presence and the maximum strength of the fibre. They found that at 65% R.H. about 50% of the fibres examined broke at a reversal point. This fraction is significantly greater than the 15% calculated on the assumption of a random occurrence of breaks along the length of the fibre, which proves that the position of reversal of the spiral direction is a weak point in the fibre. This property also offers an infallible means of identifying the cotton fibre no matter what treatment it may have had.

TESTS FOR FIBRE DAMAGE

Knowledge of some of the structural features of the cotton fibre enables us to formulate some very useful tests, which are of immediate value to the dyer and finisher. During the processing of cotton goods they are subjected to many chemical and physical treatments which may affect the fibre, such as kier boiling, bleaching, dyeing, and anti-shrink finish treatments, which may not always be perfectly controlled, and occasionally fibre damage results. Often the actual stage in processing when this has occurred is unknown, and the dyer or finisher has to ascertain rapidly the type of damage his material has suffered in order to prevent further batches from being spoilt. Most types of damage to natural cellulosic materials such as cotton affect in some way the outer, more resistant primary layer of cellulose, and most tests for fibre damage depend on the modification of this cuticle.

The Extrusion Test

The primary layer of cellulose is considerably more resistant to swelling agents of all kinds than is the secondary cellulose, and when a short length of the fibre is immersed in caustic soda of mercerising strength, considerable pressure is exerted inside the fibre, causing the interior of the fibre to be extruded wherever there is no sound cuticle to restrict it. The short lengths of normal cotton fibres shown in Fig. 20 were produced by simply cutting across a thread with two thin razor blades clamped together in a suitable holder. They were then scraped off into a drop of 18% caustic soda on a microscope slide and covered with a cover-glass. The extrusion of the contents of the interior to give this characteristic dumb-bell appearance is typical of normal fibres. Damaged fibres merely swell generally without giving this effect.

The Congo Red Test

The direct cotton dye Congo Red stains the secondary cellulose of a cotton hair much more readily than it stains the outer primary wall, and this property can be made use of in a development of the previous test. The fibre is swollen with a solution of caustic soda, causing rupture of the primary wall where damage has been inflicted, when the exposed secondary cellulose will stain heavily. Where the primary wall is intact very little staining takes place. As described by Bright in 1926[45], the sensitivity of the test can be varied by changing the concentration of the initial alkali soak. A 9% solution swells the secondary cellulose just sufficiently to close the lumen completely, very little pressure being exerted on the primary wall, and is therefore suitable for detecting mechanical or bacterial damage which has completely fractured the primary wall. Fig. 21 illustrated a few fibres which have been damaged mechanically. This type of damage is easily recognised, being characterised by the generally broken appearance of the fibres and the very patchy staining with Congo Red. Fig. 22 illustrates a few fibres taken from a material which has become mildewed from storage under damp conditions. This type of damage, in common with damage from heat, light, and chemical attack, often weakens the primary wall without destroying it completely, and an initial steep in caustic soda of about 11% concentration is generally necessary to reveal the effect. Very slight damage requires the use of 18% caustic soda. The Congo Red test has been extensively used by Clegg[46,47] working at the British Cotton Industry Research Association and by others[48] to study fibre breakdown in garments subjected to normal wear and tear.

LINEN

The linen fibre has a spiral fibrillar structure somewhat similar to that possessed by cotton, with an outer, resistant cuticle, and the extrusion and Congo Red tests may be applied similarly. For the purpose of staining the intercellular débris, of which a certain amount inevitably remains, an alcohol–concentrated hydrochloric acid solution of phloroglucinol is usually suggested[49], but, although excellent for other bast fibres, this stain is not particularly useful for linen, which contains relatively little lignocellulose. A better stain is probably Congo Red, a procedure being used similar to that employed for damaged cotton fibres. A very extensive microscopical examination of linen fibres was carried out by Searles in 1924[50], who published photomicrographs illustrating his work. Searles found also that, when a normal linen fibre was mounted in, say, 9% caustic soda, the application of pressure to the top of the cover-glass was sufficient to cause splitting of the fibre into individual fibrils. This is illustrated in Fig. 23 by means of polarised light. Chemically damaged linen is no longer capable of being split into fibrils under these conditions, and instead transverse cracking results and the fibre breaks up into a series of small rod-like components (Fig. 24).

REGENERATED CELLULOSIC FIBRES

It might easily be assumed that regenerated cellulosic fibres would provide only a limited field for microscopy, since they might be expected to possess few, if any, structural features. However, as P. H. Hermans has pointed out[51], industrial spinning methods comprise exceedingly complicated stationary processes, in which numerous

chemical and physical changes take place in a very short space of time, either simultaneously or in quick succession. They usually result in the production of a fibre which is described as consisting of a partly crystalline and not particularly well orientated core surrounded by a thin outer skin which is considerably more anisotropic. Direct cotton dyes such as Chlorantine Fast Green 5BLL (Ciba) have been used for differentially staining the skin and the core (Fig. 25), but in 1945 Morehead and Sisson [52] recommended the use of Victoria Blue B. After sectioning, staining is carried out for one hour with a 2% aqueous solution of Victoria Blue B. Staining is initially heaviest on the core, but with careful washing with solutions of water in dioxan the colour is removed comparatively rapidly from the core, the skin being left more heavily stained (Fig. 26). Hermans [53] has modified this technique by using aqueous pyridine or ethyl alcohol solutions instead of dioxan, which is poisonous. If, after carrying out this procedure, we further stain the sections with, say, Cotton Yellow CH solution, we can enhance the differentiation by obtaining the core stained yellow and the skin stained green.

The Structure of Viscose Rayon Fibres

It has been suggested by the Japanese worker Ohara [54] that the skin is further differentiated, and that there is a very thin outer skin of yet another nature. Hermans [55] has produced photomicrographs taken with low-aperture illumination, in which the image is falsified by diffraction, which bear a strong resemblance to Ohara's. However, we have obtained some slight evidence that there are, after all, three zones in some way structurally different in ordinary viscose rayon, from experiments similar to those described by Morehead and Sisson [52] and by Hermans [53] but carried out on viscose rayon previously dyed with Direct Fast Scarlet SE. Fig. 13 shows the appearance of the sections before staining. Dyeing was carried out for about two hours to give a reasonable approximation to equilibrium absorption of the dye. The centre of the fibre is dyed heavier than the outer skin. The photomicrograph shown in Fig. 9 was prepared by staining the dyed sections of Fig. 13 with Victoria Blue B and Cotton Yellow CH, by the method of Morehead and Sisson [52]. Direct Fast Scarlet SE will presumably act as a mordant for the basic dye, leading to the heavy staining in the centre of the fibre, but it is a little difficult to explain these results without assuming that there are three distinct structural regions in the fibre. Royer [56] has published phase-contrast photomicrographs of thick-skin rayon fibre cross-sections which show a similar distinct separation into three zones, but our attempts to demonstrate this with ordinary normal-stretch viscose rayon have not been too successful. The skin of viscose rayon is not sufficiently different from the core to admit of the use of tests for damage similar to those which can be used for cotton. We cannot, for example, use the Congo Red or the extrusion test on viscose rayon.

* * *

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Discussion

WEST RIDING SECTION

Mr. E. STEAD: Why do the photomicrographs of linen and jute fibres taken in polarised light show such brilliant colours?

Mr. BROWN: The cellulose molecules are arranged more or less parallel to the axis of the linen fibre and the resultant crystallinity enables the fibre to rotate the plane of polarisation of polarised light passing through it, the amount of this rotation depending on the wavelength of the incident light. Hence plane-polarised white light is split up into rays of different colours polarised in different planes. The intensity of these is differently affected by the second sheet of Polaroid, with the result that some colours predominate.

Mr. R. K. FOURNESS: Will the lecturers supply details of the home-made polariser?

Mr. BROWN: The use of Polaroid sheet in place of Nicol prisms is a distinct economy, and it is usually quite easy to make simple attachments without unduly modifying the microscope itself. Pieces of Polaroid sheet can be obtained approx. 1¼ in. square, which are quite suitable for this purpose.

Mr. FOURNESS: What method of colour photography has been used for the slides?

Mr. BROWN: We used Ektachrome B (Kodak) sheet film. This is suitable for illumination by artificial light, and can be self-processed with prepared chemicals supplied by the makers.

Mr. R. B. WHITEHEAD: Could the Sellotape system be used to "optically dissolve" completely the protein fibre in a mixture of protein and cellulosic fibres, thus leaving only the cellulosic fibre visible?

Mr. BROWN: The Sellotape strip method is designed purely to study the fine surface structure of fibres, and would not be required to distinguish between protein and cellulosic fibres. It is possible, with a mixture of protein and cellulosic fibres, to render the protein fibres almost invisible, simply by mounting in, say nitrobenzene. The difference in refractive index between cellulose and the medium would still be sufficient to make cellulosic fibres stand out fairly clearly.

Dr. F. F. ELSWORTH: I was interested in a slide showing a dry-chlorinated fibre the cortex of which had remained unstained, except at the cut ends, after immersion in a solution of Kiton Red G. It was suggested that the action of the chlorine in this process is confined to the cuticle, and that the subcuticular layer is unaffected and thus resists penetration by dyes. Have the lecturers made any similar observations on fibres treated by wet chlorination processes?

Mr. BROWN: We have carried out the double staining technique only using basic and acid dyes with dry-chlorinated fibres.

MIDLANDS SECTION

Mr. A. J. WESSON: Will the lecturers give further information on the method of "casting" fibres in resin polymers?

Mr. BROWN: For the purpose of preparing cross-sections by the plastic embedding procedure, it would be convenient first to stretch the fibres until they were arranged parallel by means of a suitable cardboard holder, and then to introduce them into a solution of plastic monomer and plasticiser. A mixture of 10 parts of methyl methacrylate to 3 parts of methyl phthaloyl ethyl glycollate has been used successfully. Benzoyl peroxide (0·5%) is added immediately before use to initiate polymerisation, and when fully polymerised the block can be trimmed down prior to sectioning in the usual manner. Examination is usually possible without removal of the medium.

Mr. A. P. KERSHAW: Two of the slides shown have a magnification of 20,000. Have any higher figures been achieved?

Mr. BROWN: Most modern commercial electron microscopes have a resolution of the order of 50–100 A., which enables a useful magnification of about 25,000 to be obtained. With selected instruments and careful technique a resolution of the order 20 A. has been claimed. This would correspond to a useful magnification of the order of 100,000. In theory, of course, the ultimate possible resolution of the electron microscope is of the order of 0·1 A., giving a useful magnification of the order of 25,000,000, but in practice the instrument is very far from perfect, having an extremely small numerical aperture, for instance.

Mr. A. W. CARPENTER: The lecturers have shown the Victoria Blue B staining technique to differentiate between normal and shrink-resisted wool. Are any characteristic differences exhibited as between (*a*) acid chlorination, (*b*) alkaline chlorination, (*c*) alcoholic potash treatment?

Mr. BROWN: The Victoria Blue B staining with acid- and alkali-chlorinated wool is very similar, and could not be used as a method of distinguishing between these two treatments. Staining on wools treated with alcoholic potash solution is similar, but usually more general and heavier. There are usually a greater proportion of fibres showing very heavy localised staining, indicating considerable damage.

Mr. R. A. GALE: The use of various liquid media for slide mounting has been discussed. Can any useful hints be given on the preparation of slides of a more permanent nature?

Mr. BROWN: Glycerol jelly is a suitable medium of low refractive index for making permanent mounts. It is prepared from gelatin and glycerol with a small amount of phenol as a preservative, and can be diluted with water to any required consistency. It is necessary to melt the jelly immediately before use. After mounting, the cover glass should be sealed and ringed with a solution of cellulose nitrate lacquer. Canada balsam is often used in pathology as a permanent mount, and it has the advantage of being non-aqueous, although it is prone to yellowing with age and is tedious to use. There is a useful neutral

mountant called *Euparal* (Flatters & Garnett Ltd.), which is claimed to be easier to use and more effective than Canada balsam.

Miss Y. MALLION: Will the lecturers give further details on the mounting of fibres with Sellotape?

Mr. BROWN: The Sellotape strip method was designed to reveal small structural differences in the surfaces of fibres, and hence it is extremely useful for the natural protein fibres. There are few other fibres which possess any pronounced surface structure. With regard to the technique, the usual mistake is to attempt to embed too many fibres on the same slide, with the result that pressure is applied unevenly and only a few of the fibres become embedded to the correct extent. A little practice soon reveals how much pressure to apply. The preparation should be examined under the microscope without delay, since the fibres sometimes tend to part from the gum of the Sellotape.

Miss MALLION: What is the best mounting medium for the examination of nylon?

Mr. BROWN: For normal routine examination, water would appear to be quite satisfactory as a mountant for nylon.

Mr. G. E. KEY: Have the lecturers any information on the use of the phase microscope for the study of surface effects?

Mr. BROWN: The phase-contrast microscope is useful for demonstrating fine structure resulting from small differences in optical path thickness which cannot be observed directly by, say, staining. In this respect its usefulness is limited, and we are not aware of any application to the examination of fibre surface structure. However, the phase-contrast microscope has been used with some success to examine the distribution of synthetic resins applied to the outside surface of wool fibres.

Mr. J. C. HAWLEY: Can a staining technique be used to distinguish mildew damage from bacterial attack on cotton?

Mr. BROWN: The Congo Red test is of some assistance in this case, although whether or not a distinction can be made depends on what further treatment a material has undergone after suffering attack. Generally, bacterial attack is extremely severe on a few fibres, resulting in their complete destruction, although neighbouring fibres may be completely undamaged. Mildew attack is usually more general, and many more fibres are affected, although the actual fibre damage may be less. It is sometimes possible to confirm mildew by the recognition of mildew hyphae on the fibre. These stain heavily with direct cotton dyes.

Mr. HAWLEY: Can microscopic examination be used for determining the type of dye used?

Mr. BROWN: We know of no method of identifying dyes by the direct microscopical examination of fibres taken from dyed material.

Mr. H. C. OLPIN: If the dyed fibre is examined under polarised light, dyes of long-chain structure would give greater polarisation than dyes (e.g. azoics) having small molecules, but positive results cannot be guaranteed.

Mr. BROWN: It is well known that some direct cotton dyes, more particularly those with a straight-chain structure, are preferentially orientated in the direction of the fibre length, and that the resultant dichroism can be demonstrated with polarised light. Some azoic dyes also exhibit dichroism, and in some cases the plane of maximum absorption of light is rotated through 90° as compared with dyeings of direct cotton dyes. This indicates that with azoic dyes the absorbing conjugated chain must be arranged perpendicular to the axis of the cellulose molecules.

Mr. E. FELL: In the opinion of the lecturers, are the slides submitted from Gothenburg a satisfactory confirmation of Prof. Gralén's hypothesis?

Mr. BROWN: The existence of a thin exterior membrane on keratinous substances was postulated before the isolation of the epicuticle from wool by Prof. Gralén. The several methods of isolation used, the chemical properties, and the dimensions of this membrane make it impossible to doubt that it originates from the surface of the cuticle cells of the fibre. In addition, there is the accumulating evidence of the existence of membranes on other keratinous substances and the remarkable similarity of these to that isolated from wool. As regards the exocuticle, the electron-micrographic evidence is less convincing, but the action of halogens in the Allwörden test makes it necessary to postulate some kind of layer of a different chemical nature beneath the epicuticle.

Mr. J. A. DONOVAN: Can any technique be devised to determine differences in degree of mercerisation of cotton fibres?

Mr. BROWN: When cotton fibres are mercerised the cross-sectional shape becomes more or less circular and the lumen contracts almost completely, while the fibre convolutions are removed. The characteristics of individual fibres vary considerably, however, and it is not possible to recognise the degree of mercerisation by simple observation. The best and most accurate method involves cutting the fibres into lengths of the order of 0·01 in. by means of two razor blades clamped together and determining the percentage number of convoluted fibre lengths. Among staining tests which have been suggested is one described by Kinhead [57] which uses Methylene Blue and iodine. Greater reliability than in previous tests is claimed for this stain.

PUBLICATIONS SPONSORED BY THE SOCIETY'S FASTNESS TESTS CO-ORDINATING COMMITTEE— XI

The Development of the Geometric Grey Scales for Fastness Assessment

FASTNESS TESTS CO-ORDINATING COMMITTEE

The methods which have been used since 1914 for assessing the results of fastness tests are described in detail. The most recent method is to use grey scales for this purpose, and scales equal in spacing are now being distributed by both E.C.E. and the Society. This method may well become standard all over the world.

INTRODUCTION

When a coloured pattern has been subjected to a fastness test, it is usually necessary to record information which will enable interested persons to learn how the pattern has withstood the test. The simplest method of doing this is to use descriptive terms such as "slightly altered" and "heavily stained", as were used in the original *Colour Index* [1]. This method is, however, subjective and therefore lacking in precision, as different people will attach different meanings to the qualitative terms employed; mere replacement of such terms by numerical ratings does not, of course, effect any improvement in precision, and various organisations during the past forty years have devised improved assessment methods. Various methods have been developed and these can be subdivided as follows—

(*a*) Pass–fail tests
(*b*) Dyed standards
(*c*) Fixed standards.

To-day, the ultimate development in the use of fixed standards, i.e. grey scales, is likely to become the standard method in many countries. The development of these grey scales has been characterised by international co-operation in the field of fastness testing since 1948, and the purpose of this review is to trace this development and to record the part played by the various participating organisations. This can best be done by considering each of the methods used in some detail.

(*a*) *Pass–Fail Tests*

In this method, the pattern under test is subjected to a series of tests of increasing severity, and is deemed to pass each if there has been no alteration in the pattern; the fastness rating is the number of the most severe test that it passes. The main objections to this method are that it does not enable small but important differentiations to be made and that as many as four different tests may have to be made in order to determine the fastness to any particular agency. The method is valid in Great Britain (for washing tests only) and the U.S.A., and may well be eventually abandoned in view of recent developments in international standardisation.

(*b*) *Dyed Standards*

This method was devised by the Deutsche Echtheitskommission (D.E.K.), a body formed as long ago as 1911. It was first published in 1914 as a manual, *Verfahren, Normen und Typen* [2], and the original proposals were modified and improved during the years 1914–1939, with the result that, in all, eight editions of this well known manual were published.

The method consisted in subjecting to the test, together with the patterns under test, three dyed standards representing the major fastness grades 1, 3 and 5. When the test was complete, the tested patterns were compared with the effects produced on the dyed standards and assessed accordingly, the intermediate grades 2 and 4 being permitted.

This method was finally abandoned, partly because it was too complicated for general use—over 140 dyed standards were needed for the pre-war fibres wool, cotton, silk, and acetate rayon—and partly because it did not separate the effect produced on the pattern from the staining of adjacent undyed material.

(*c*) *Fixed Standards*

The idea of using fixed standards was put forward completely independently in three different countries, and this fact seems to show that the method is the only rational solution to the problem of assessment.

In this method the patterns and the adjacent undyed materials are compared after testing with a series of fixed standards which illustrate the various fastness grades. Unlike the dyed standards of the D.E.K., however, these fixed standards are not produced each time the test is carried out, but are separately prepared once only, and then not by a fastness test, but by dyeing, printing, or similar methods. Thus for the assessment of staining, a series of five patterns ranging from white (to illustrate grade 5) to a medium depth (grade 1) would be used.

The first proposal to use fixed standards was made in 1939 by the late L. A. Lantz, who was at that time a member of the Society's Washing Fastness Sub-Committee. In January 1940, fixed standards in various hues for assessing both loss in depth and degree of staining which he had prepared, were considered by the parent Fastness Tests Committee of the Society; acceptance was, however, deferred, as the tests themselves were at that time under revision. The idea of using fixed standards was subsequently conceived and developed quite independently by—

(i) Imperial Chemical Industries Ltd. in 1942–1943 (Great Britain) [3]
(ii) J. R. Geigy AG. in 1943–1944 (Switzerland) [4]
(iii) U.S. Quartermaster Corps in 1945 (U.S.A.) [5]

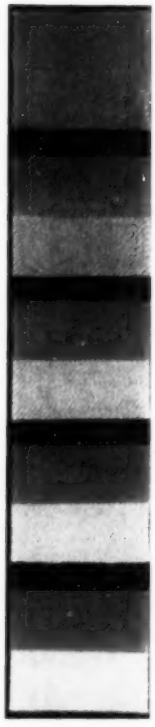


FIG. 1

S.D.C. Grey Scale
No. 2

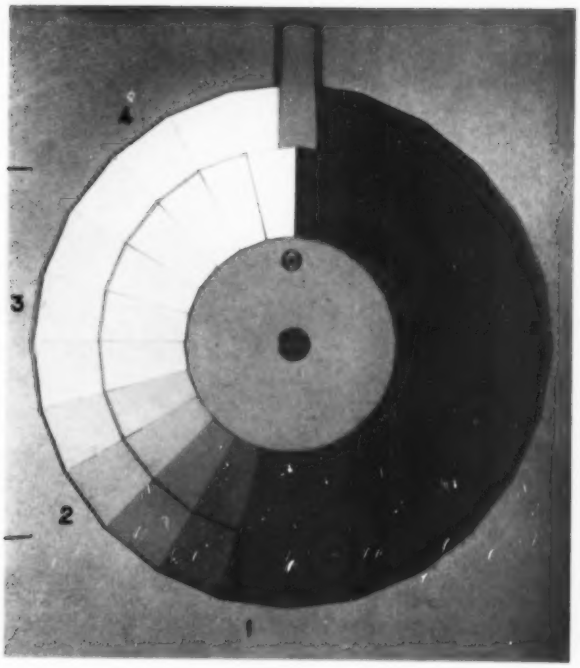


FIG. 2A

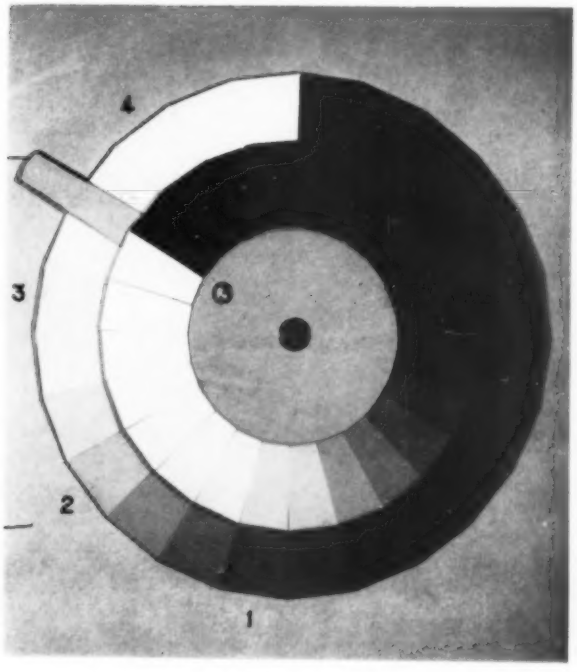


FIG. 2B

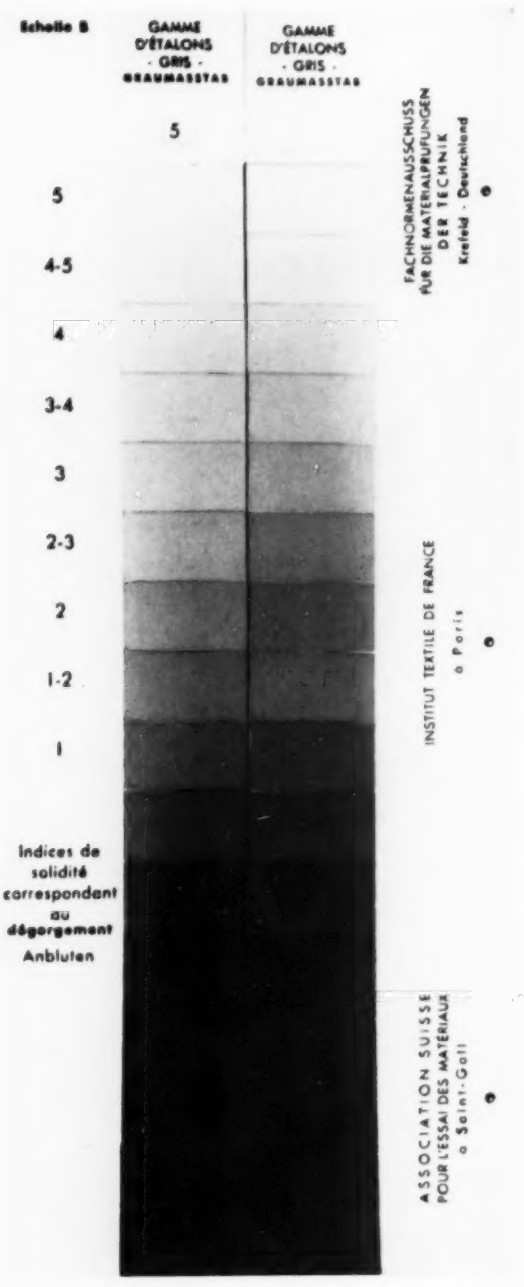


Fig. 3 — Sixteen-step Franco-Swiss Scale

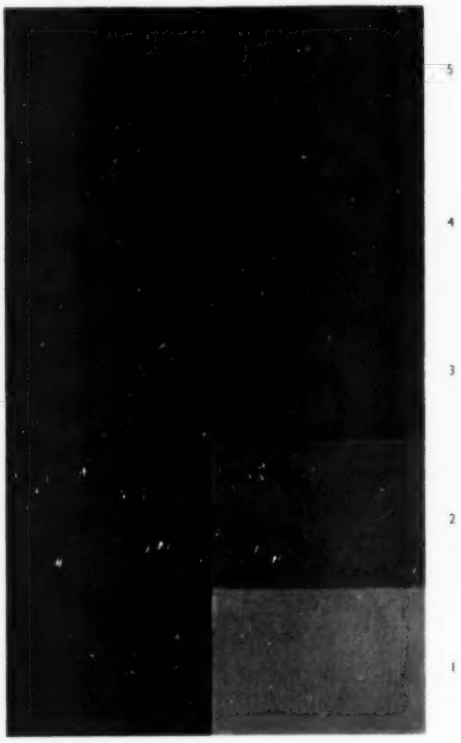


Fig. 4 — Original Geometric Grey Scale

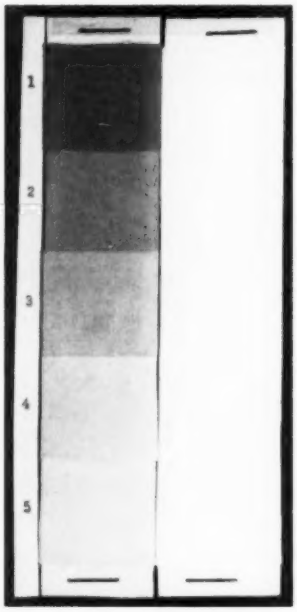


Fig. 7 — Original S.D.C. Staining Scale

Type A

Type B

Type C

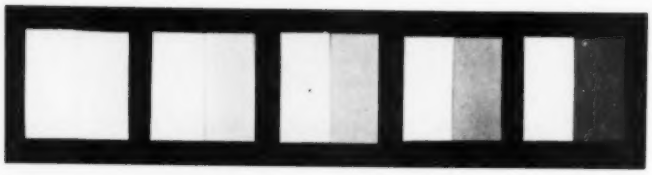
FIG. 5 Geometric Grey Scales - I.S.O. Prototypes

E.C.E. Grey Scale

I.S.O. Prototype B

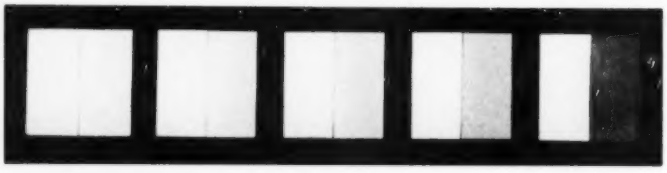
S.D.C. Grey Scale

FIG. 6 Geometric Grey Scales for Assessing the Effect on the Pattern



Unequally spaced Scale

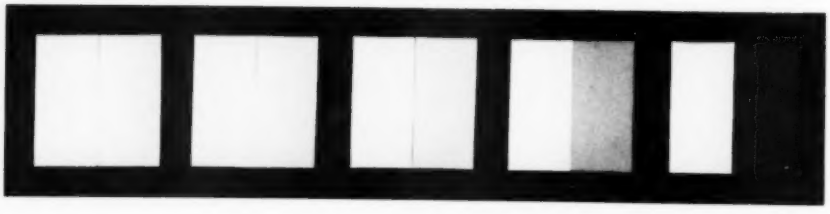
Arithmetic Scale



Geometric Scale

FIG. 8—Staining Scales S.D.C. Prototypes

E.C.E.



S.D.C.

FIG. 9—Geometric Staining Scales now available

The American development was restricted, as it was applicable only to the assessment of the results of rubbing tests; it is, however, of particular interest, as it was the first time a single grey scale was intended to be used for the assessment of patterns of every hue. The original proposal was ahead of its time, as it was subsequently modified by the Research Committee of the A.A.T.C.C., and ended up as the *Transference of Color Chart* [6] in six different hues!

The systems independently developed by ICI and Geigy were similar and quite complex, as not only were loss-in-depth and staining scales prepared in different hues, but both concerns prepared change-in-hue scales; thus in the ICI system sixty coloured standards were required for assessing the effect on the pattern, and even then no attempt was made to illustrate combinations of loss in depth and change in hue. Being specifically designed for determining the fastness properties of dyes as distinct from dyed patterns, these systems were concerned only with patterns dyed to a standard depth; for general-purpose testing, therefore, where patterns of every possible depth are concerned, these proposals are not suitable, and it is easy to see that to extend their scope would require the preparation of such a large number of scales as to make the proposal quite impracticable.

The most recent development of the concept of fixed standards has therefore been the replacement of these scales in many hues by only two, both grey, one for assessing the effect on the pattern and the other for determining the degree of staining. As each was developed at different times, it is convenient to consider them separately, although they are obviously complementary.

Grey Scales for Assessing the Effect on the Pattern

The first proposal to use a grey scale for assessing strength differences between patterns, irrespective of hue, was made by P. W. Cunliffe, who in March 1947 produced the precursor of the S.D.C. Grey Scale No. 2 (Fig. 1). This was designed primarily for assessing the results of migration tests on wool dyes [7], but this problem is, of course, fundamentally the same as that of assessing loss in depth in fastness testing.

The second grey scale which appeared—the first to be specifically intended for use in fastness assessment—was designed by J. Weibel of the Swiss organisation E.M.P.A.[8]. His primary intention was to produce a scale which could be used for assessing loss in depth of patterns of any original depth whatsoever. This was ingeniously achieved by constructing a scale which extended in twenty-four visually equal steps from black to white. Its method of use was to determine which grey areas equalled the depth of the pattern before and after testing; the interval between them was inversely proportional to the fastness rating. For ease in manipulation, two identical scales were mounted concentrically (Fig. 2A), and on rotation to bring the two selected grey areas adjacent, the fastness rating appeared in the window provided (Fig. 2B).

The use of this scale was then investigated statistically by J.-P. Niederhauser [9], who found that the variations between observers who had only slight training in assessment were serious, and that some found it impossible to use the scale for yellows and oranges; the scale itself was also found to be unequally spaced—a defect of the Weber–Fechner Law employed in its construction. He therefore prepared a new sixteen-step grey scale based on the Munsell Colour Atlas, the two component scales being arranged linearly instead of concentrically (Fig. 3), and he intended to supplement this with scales in other hues where necessary; the choice of the Munsell system was partly due to the ease with which coloured scales, visually equal to the grey scale, could be prepared.

This method of use was, however, fundamentally different from that proposed by Cunliffe for S.D.C. Grey Scale No. 2, which was to assess the *contrast* between the patterns by comparison with the five fixed contrasts illustrated on the Grey Scale. Both these scales were exhibited at the first international fastness conference held on the Continent (Basle, 1951), and Cunliffe, by then Chairman of the Fastness Tests Co-ordinating Committee, mentioned that this committee thought the contrast comparison method to be preferable.

In one other important respect, however, the S.D.C. scale clearly resembled the sixteen-step Franco-Swiss scale—the spacing was very similar.

In order to compare grey scales, it is better to measure the spacing rather than to rely on visual comparison. This is quite easily done by plotting the reflectance curves, e.g. by means of the G.E.C. recording spectrophotometer (popularly known as the "Hardy"). These curves can then be integrated to obtain C.I.E. data, and from these, the colour difference can be calculated by one or other of the several formulae available. The one which has been used for grey scales is the Adams "chromatic value" formula—

$$1E_A = 40\{[0{\cdot}23\,1V_y]^2 + [\tfrac{1}{2}(V_x - V_y)]^2 + [0{\cdot}4(V_z - V_y)]^2\}^{0.5}$$

This formula expresses the colour differences in N.B.S. (National Bureau of Standards) units, one N.B.S. unit being defined as the smallest difference which is of commercial significance; this unit has also been termed a "just perceptible step", but this is misleading, as trained colourists can easily perceive much smaller colour differences.

The comparison of the two scales is given in Table I.

Table I

Grade	(N.B.S. units) Grey Scales	
	16-step Swiss	S.D.C. No. 2
5	0	0
4	4·2	4·9
3	8·4	9·9
2	12·6	15·1
1	16·8	17·5*

* The bulk preparation of this scale gave too great a depth for Grade 1, but because of its relative unimportance, this was not immediately corrected.

Shortly after the Basle meeting, K. McLaren pointed out [10] that equally spaced scales— whose contrasts increased in arithmetical progression— were unsuitable for assessing the effect on the pattern, and recommended that such scales should be spaced geometrically. The fundamental difference between the equally spaced and the geometric scales is given in Table II.

Table II

Grade	Contrast (arbitrary units) Equally Spaced	Geometric
5	0	0
4	n	n
3	$2n$	$2n$
2	$3n$	$4n$
1	$4n$	$8n$

The superiority of the geometric scale lies in the fact that it is closely spaced in the 5, 4, 3 region and widely spaced in the 2, 1 region.

This proposal was accepted by the F.T.C.C., and the first geometric grey scale, spaced 0, 2·5, 5, 10, 20 N.B.S. units (Fig. 4), was discussed at the second meeting of the Colour Fastness Sub-committee of the International Organisation for Standardisation (ISO/TC 38/SC 1) held in Bournemouth in June 1951. At this meeting, which included delegates from France and Switzerland and observers from Germany, which at that time was not a member of I.S.O., it was agreed that grey scales for assessing loss in depth should be spaced geometrically and that the contrast-comparison method of using them should be employed. A decision on the actual spacing of the scale was deferred, however, and the F.T.C.C. was asked to prepare two other scales, which, with the original, would be discussed separately. The F.T.C.C. therefore sent to the European and American fastness committees these three geometric grey scales, together with another which was of potential interest; the actual spacings are given in Table III, and the three additional scales are illustrated in Fig. 5.

Table III

Grade	Geometric Grey Scales (N.B.S. units) Original (× 2)	A (× 2)	B (× 2)	C (× 2·5)
5	0	0	0	0
4	2·5	2	1·5	1·5
3	5	4	3	3·75
2	10	8	6	9·5
1	20	16	12	24

In October 1951 the F.T.C.C. was informed that the French–Swiss–German fastness tests co-ordinating committee preferred Scale B. In February 1952 the committee received a grey scale which had been prepared by P. Rabe of the re-formed D.E.K. on behalf of the Continental committees, now formally united as E.C.E.; this scale was of the same format as S.D.C. Grey Scale No. 2.

This scale was not, however, generally issued, and somewhat later Rabe prepared another, which is now freely available; this was found to be very similar in spacing to the preferred Scale B, which had, of course, been accepted by E.C.E. as the prototype.

The F.T.C.C. therefore prepared a document which, in essence, specified that grey scales for use in assessing the effect on the pattern in fastness testing should be spaced 0, 1·5, 3, 6, 12 N.B.S. units. This document was submitted to the third I.S.O. meeting on colour fastness (New York, November 1952), which was attended by delegates from France, Switzerland, and Germany— now a member of I.S.O.; this proposal was unanimously accepted as a Tentative International Standard.

At this time, the F.T.C.C. was also preparing grey scales in bulk equal in spacing to Type B, so that today both E.C.E. and S.D.C. scales are freely available; these are illustrated, together with their prototype, in Fig. 6, and are identical in the only important aspect, i.e. spacing; both are now being examined by the A.A.T.C.C.

The only intentional difference between them is that the E.C.E. scale is made from dyed cotton and the S.D.C. scale from pigment-coated card.

There is one further development in the use of the geometric grey scale which is of considerable importance. The method of using the original twenty-four- and sixteen-step Swiss scales obviously limited their use to the assessment of loss in depth; when the E.C.E. adopted the contrast-comparison method and the five-step scale, it was still restricted to this purpose. For patterns which changed in hue, a subjective method was used, e.g. 1R slightly redder, 3G much yellower, which, of course, in the absence of illustration, is inevitably vague. McLaren, however, showed statistically [11] that the contrast-comparison method can be used also for assessing change in hue or brightness, and, in fact, can be used for assessing any combination of changes whatever. A proposal to use the scale for this purpose was therefore also made by the F.T.C.C. at the New York Meeting of the I.S.O., and was also accepted as a Tentative International Test.

Grey Scales for Determining Staining

The preparation of fixed standards for determining the degree of staining is very much simpler than the preparation of fixed standards for assessing the effect on the pattern, as the problem is complicated neither by changes in hue nor by differences in depth of the original material. Consequently, the use of fixed standards in different hues is quite a feasible proposition.

In 1946–47 Marney and Davies [12] investigated this problem to decide which of three possible systems should be adopted. These were— (i) to use the A.A.T.C.C. Transference of Color Chart, which is in six hues; (ii) to increase the number of hues to nine or ten; or (iii) to reduce the number to one (grey). The answer was obtained by statistically analysing over three thousand assessments made using the ICI fixed standards in nine hues and the grey section of the A.A.T.C.C. chart, matched on cotton and augmented by the addition of undyed material so that the contrast-comparison method could be employed. This analysis proved conclusively that a grey scale was not significantly inferior to the nine-hue ICI system, and subsequent

developments have therefore been entirely confined to grey scales.

The first single grey scale for staining assessment other than the restricted American method already mentioned was produced experimentally by the Society in 1948 [13] and is illustrated in Fig. 7. This differed from the grey scales which had been included in the earlier multi-hue systems of the dye manufacturers in the following respects—

(i) S.D.C. grade 5 represented the maximum degree of staining which is allowed to be described as "virtually unstained" and the A.A.T.C.C. grade 5, although not illustrated, was similarly described as "practically unstained"; in the ICI and Geigy systems, however, grade 5 was completely unstained, any just perceptible staining being rated 4–5.

(ii) S.D.C. grades 4 and 3 were identical with A.A.T.C.C. grades 4 and 3.

(iii) S.D.C. grades 2 and 1 were lighter than those of the A.A.T.C.C., as the S.D.C. scale was intended to be equally spaced according to Fechner's law, while the A.A.T.C.C. grey scale was equally spaced according to the Munsell Colour Atlas. This difference was, however, unimportant, as any degree of staining greater than grade 3 is so serious that any minor differences in assessment would be insignificant.

The next staining scale proposal came from the Swiss, as certain of the greys in the twenty-four-step scale published in 1949 were intended for use in staining assessment. The same was also true in the case of the subsequent sixteen-step Franco–Swiss scale, and as this was based on the Munsell Colour Atlas it was possible for these greys to be identical with those of the A.A.T.C.C. Transference of Color Chart, i.e. Munsell values of 9, 8, 7 and 6.

When McLaren proposed that scales for assessing the effect on the pattern should be geometrically spaced, he also realised that this applied equally to staining scales; the F.T.C.C. subsequently, in August 1951, sent one such scale to E.C.E. and to the A.A.T.C.C. for comment. The actual spacing selected for the scale is given in Table IV, the

Table IV

Grade	N.B.S. Units from White
5	0
4	7
3	14
3	28
1	56

particular choice being governed by the desire to make the important grade 4 equal to the A.A.T.C.C. grade 4; unfortunately, this particular construction resulted in a scale which was too widely spaced. In spite of this defect, however, it was sent to the Continental committees for the prime purpose of obtaining agreement on the adoption of the geometric principle; the accompanying memoranda pointed out its defect and mentioned that a more suitable geometric spacing could be selected if necessary. The Continental committees, however, preferred an equally spaced or arithmetic scale, and one such scale had, in fact, been received by the F.T.C.C. at this time from Rabe of the German committee. Unfortunately, this had been prepared on unbleached wool, and the yellow colour of the higher grades was somewhat objectionable. In December 1951, the British committee therefore prepared a similarly spaced, i.e. arithmetic, scale on bleached cotton, and submitted this to the Continental committees.

The F.T.C.C. were not, however, satisfied that this arithmetic scale was entirely satisfactory, and in June 1952, therefore, they considered in detail the question of suitable spacing for the staining scale [14]. The grey section of the A.A.T.C.C. scale consisted of four Munsell chips (Table V). The

Table V

Grade	Munsell Value
4	9
3	8
2	7
1	6

problem was to make a series of five contrasts by including one of zero contrast and using as many of the existing four A.A.T.C.C. standards as possible. The theoretical Munsell grey series ranges from white (Munsell value 10) to black (Munsell value 0) in ten visually equally spaced steps. The simple solution, therefore, would be to add patterns of Munsell value 10 (for zero contrast), which would produce an equally spaced scale; unfortunately, however, patterns of this value (100·0% reflectance) cannot be produced commercially, the maximum value obtainable being about 9·6 (90% reflectance). Taking everything into consideration, therefore, the Committee considered that there were three possible solutions—

(*a*) To take the existing A.A.T.C.C. standards for grades 1, 2, 3 and 4 together with a grade 5 of Munsell value 9·5; this scale is unevenly spaced.

(*b*) To make grade 1 equal to A.A.T.C.C. grade 1 (i.e. Munsell value 6), to make grade 5 equal to Munsell value 9·5, and to space the intermediate grades equally between them; this scale is spaced *arithmetically*.

(*c*) To make grade 1 equal to A.A.T.C.C. grade 1 (i.e. Munsell value 6), to make grade 5 equal to Munsell value 9·5, and to space the intermediate grades *geometrically*.

Scales were therefore prepared to illustrate these three possibilities (Fig. 8), and the Committee unanimously selected the geometric staining scale. The colorimetric data for this scale are given in Table VI, from which it will be observed that

Table VI

Grade	Theoretical (N.B.S. units from white)	Actual (N.B.S. units from white)	Munsell Value
5	0	0	9·38
4	4	4·2	8·98
3	8	7·8	8·60
2	16	15·8	7·76
1	32	31·8	6·02

grade 4 as well as grade 1 is identical with the corresponding A.A.T.C.C. grade; this was unexpected, but is another reason why this scale is the most attractive. The reason why the spacing differs so greatly from that of the first geometric scale (Table IV), which was deliberately constructed so as to make grade 4 equal to the A.A.T.C.C. grade 4, is that the material used for grade 5 of the first scale was bleached cotton limbric with a Munsell value of 9·66; in the second scale, pigment-coated white cards were used with a Munsell value of only 9·38. The F.T.C.C. now felt that, if no other scale had ever been proposed, the geometric scale would still have been selected out of all the possible alternatives, as its spacing closely illustrated the Committee's view on each grade.

At the same time as Rabe prepared the experimental grey scale for assessing the effect on the pattern, he prepared also an experimental staining scale. This was measured by Niederhauser, and the results are given in Table VII, from which it

Table VII

Grade	N.B.S. Units from White
5	0
4	2·5
3	4·4
2	10·2
1	19·3

will be observed that this scale is geometrically spaced. It was not generally issued, however, but when the subsequent scale for assessing the effect on the pattern appeared, it was accompanied by a staining scale. Patterns of the cloth used for this scale were received from Rabe, and the spacing is given in Table VIII, which shows that this spacing is practically identical with that favoured by the F.T.C.C. (Table VI). The E.C.E. had, therefore,

Table VIII

Grade	N.B.S. Units from White
5	0
4	3·6
3	8·5
2	15·0
1	26·0

eventually accepted the S.D.C. proposal that the staining scale should be geometrically spaced; they had, however, reached the same conclusion with regard to the magnitude of the spacing quite independently.

The F.T.C.C. prepared a document for submission to the New York I.S.O. meeting which described the three possible spacings, i.e. unequal, arithmetic, and geometric, and the preferred geometric scale was accepted as a Tentative International Standard.

As in the case of the final scale for assessing the effect on the pattern, both the S.D.C. and the E.C.E. are distributing geometric staining scales (Fig. 9) which are identical in spacing; both are now being examined by the A.A.T.C.C.

Future Possibilities

Although prophecy is extremely dangerous, the development of the geometric grey scales has been such that it is probable that they will enjoy some degree of permanence. Since 1914, with one exception, all possible methods of assessing the results of fastness tests have been fully explored, with the result that only one, i.e. the use of grey scales whose spacing has been defined, has been found to be generally suitable.

The only method which might conceivably displace the use of geometric grey scales is for the colour difference between the original and tested patterns (or between stained and unstained material) to be determined by optical methods. One apparent advantage of such methods is that they eliminate the human element and thus should result in greater reliability and the absence of differences of opinion.

The Fastness Tests Co-ordinating Committee, however, has had sufficient experience of colour-difference formulæ to be convinced that their use in fastness testing is dangerous; two typical examples illustrate their unreliability—

Godlove [15] found, using the Adams chromatic value formula, that the British light fastness standard 5 was not as fast as standard 4, and Ricketts [16], using the same method, was able to confirm this. Ricketts, however, was able to prove, by statistical analysis of the visual assessments of ten observers, that standard 5 is twice as fast as standard 4 (as intended), and that both the Adams formula and the "improved colour-difference formula" of Godlove [17] give results completely contrary to visual observation.

The other example concerns the standard "appreciable colour loss" obtained by exposing A.A.T.C.C. Light Fastness Standard 4 for 20 standard fading hours. By measurement with both Hunter Color Difference Meter and the General Electric Recording Spectrophotometer, this is a contrast of 2·4 N.B.S. units. Forty observers were asked to assess this contrast visually, using the geometric grey scale in the approved manner: their average assessment was 1·35 N.B.S. units; only three rated it visually equal to the measured value, and no-one rated it above.

The unreliability of these colour-difference formulæ, however, does not in any way lessen their inestimable value for comparing grey scales and particularly for establishing grey scales of identical spacing, which are intended to be used for visual assessment. But it cannot however be assumed, e.g. that a *measured* colour difference of 3 N.B.S. units between two patterns will be *visually* equal to the corresponding 3 N.B.S. units of grade 3 on the Geometric Grey Scale for Assessing the Effect on the Pattern.

(*Received 1st October 1953*)

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PUBLICATIONS SPONSORED BY THE SOCIETY'S FASTNESS TESTS CO-ORDINATING COMMITTEE—XII

Proposed International Tests for Colour Fastness of Textiles

International Organisation for Standardisation
Technical Committee on Textiles
Subcommittee on Colour Fastness Tests

Foreword to I.S.O. Tests

The International Organisation for Standardisation (I.S.O.) was formed in 1947 and arose from the United Nations Standardisation Co-ordinating Commission, which was set up by the United Nations Organisation during the war.

Membership is open to the officially recognised standard bodies in any country, and at present comprises some thirty countries with more than seventy technical committees dealing with a diverse range of subjects. I.S.O. Technical Committee 38, of which the Secretariat is entrusted to the United Kingdom, deals with textiles, and the U.K. membership of this committee is vested in the British Standards Institution, which therefore is responsible not only for the international secretarial activity, but also for the presentation of the U.K. point of view.

Technical Committee 38 has set up many different subcommittees, of which Subcommittee No. 1 deals specifically with colour fastness testing, the Secretariat being shared jointly by the U.S.A. and the U.K. These countries, through their standards organisations, have arranged that the technical and secretarial work for the subcommittee should be conducted by the American Association of Textile Chemists and Colorists and the Society of Dyers and Colourists. These organisations thus act as the focal points for the conduct of international negotiations, and collaborate with the other international bodies concerned through the American Standards Association and the British Standards Institution.

Colour fastness testing was discussed in general terms at the first meeting of the I.S.O. Textiles Committee at Buxton in 1948, and subsequently by correspondence between the principal countries concerned and by personal contact. At the second meeting of the Textiles Committee held at Bournemouth in 1951 there were several sessions of the No. 1 Subcommittee, at which a range of fastness tests was discussed and a wide measure of agreement on them was reached. In November 1952 a series of meetings of I.S.O. Subcommittee No. 1 was held in New York, when further progress was made.

The tests which are now put forward as tentative international standards are the outcome of continuous negotiation and co-operation between the European Continental fastness committees, the A.A.T.C.C., and the S.D.C. All these bodies have been very active during the last few years, and each of the three groups has carried out independent investigations not only with the immediate object of establishing national tests but with the wider intention of securing agreement on a series of tests which could be accepted throughout the world.

The Continental group now comprises most of the countries in Continental Europe. Following a period of close collaboration, the fastness committees of Germany, France, and Switzerland called an international meeting at Basle in February 1951 with the object of establishing agreed fastness tests for the European continent. At a second meeting in Paris in April 1952 it was agreed to form the E.C.E. (Europäisch-Continentale Echtheits-Convention or Groupement d'Études Continental-Européen pour la Solidité des Teintures et Impressions).

A third international meeting was held under the auspices of the E.C.E. at Bad Kissingen, Germany, in April 1953. The Society was represented at all three conferences by the Chairman and other members of the F.T.C.C., who, while attending only as observers, were nevertheless able to express the views of the Society, and these were given full consideration by the Continental members. Discussions were also held during the conferences on the proposals which were under consideration by I.S.O. At the Bad Kissingen conference the E.C.E. resolved that the tests which they had worked out should be adopted immediately by their members but it was made clear that they would be superseded by I.S.O. tests as soon as these had been formally accepted by I.S.O. Technical Committee 38.

Collaboration with the A.A.T.C.C. has been a happy feature of the last few years, though for geographical reasons it has had to rely mainly on correspondence. The A.A.T.C.C. has made a special feature of fastness tests over a long period

and has established tests for fastness to a large number of agencies; as with the E.C.E. and the Society, they are most anxious to see the establishment of international tests.

The tests which are now put forward as tentative international standards are published for information and comment. They do not cover all the agencies for which fastness tests are required, but others are under discussion by the various national bodies concerned. Additional tests for fastness to washing are also under consideration.

The draft of the tests has been prepared by the U.S.A. Secretariat following the New York meeting, and is published here with minor changes of spelling.

The Society will adopt the international tests as soon as they have been officially accepted by I.S.O. They are being issued simultaneously as draft British Standards by agreement between the Society and the British Standards Institution.

List of Documents on Colour Fastness

ISO/TC 38/SC 1 Numbers 31 to 58 inclusive
(25th August 1953)

Title	SC 1 No.
Colour Fastness of Textiles: General principles of testing	31
Colour Fastness: Grey scale for evaluating change in colour	32
Colour Fastness: Grey scale for evaluating staining	33
Colour Fastness to—	
Acid spotting	34
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ISO/TC 38/SC 1/31
Colour Fastness of Textiles: General Principles of Testing

NOTE—This draft, based on ISO/TC 38/SC 1/2, 1st September 1952, has been prepared as a result of the discussions of the Subcommittee in New York on 10–12th November 1952. Following the meetings the draft was circulated in preliminary form to the delegates for comment and criticism and this final form includes the suggestions which have been received. It is now before the Subcommittee for discussion at its next meeting.

1. INTRODUCTION

1.1 This standard provides general information about the methods for testing colour fastness of textiles for the guidance of users. The uses and limitations of the methods are pointed out, several terms are defined, an outline of the form of the methods is given, and the content of each section discussed. Procedures common to a number of the methods are discussed briefly.

1.2 By *colour fastness* is meant the resistance of the colour of textiles to the different agencies to which textiles may be exposed during manufacture and subsequent use.

1.3 The methods may be used not only for evaluating colour fastness of textiles, but also for evaluating colour fastness of dyestuffs. When so used, the dyestuff is applied to the textile in specified depths of colour by stated procedures and the textile is then tested in the usual way.

1.4 Each method is concerned with colour fastness to a single agency. No directions are given for testing fastness to a succession of agencies as the agencies of interest in a particular case and the order of application will vary.

1.5 The conditions in the tests have been chosen to correspond closely to treatments usually employed in manufacture and to conditions of ordinary use. At the same time they have been kept as simple and reproducible as possible. As the tests cannot hope to duplicate all of the conditions under which textiles are processed or used, the fastness ratings must be interpreted according to the particular needs of each user. They provide, however, a common basis for testing and reporting colour fastness.

2. GENERAL PRINCIPLE

2.1 A specimen of the textile to be tested, with undyed cloth attached if staining is to be rated, is subjected to the action of the agency in question. The extent of any change in colour and of any staining of the undyed cloth are evaluated and expressed in fastness numbers.

3. OUTLINE OF FORM OF THE METHODS

3.1 The headings of the principal sections of the methods follow—

Purpose and scope
Principle
Apparatus and reagents
Specimen
Procedure
Report
Notes

4. PURPOSE AND SCOPE

4.1 Under this heading in each method are given the intended use of the method, its limitations, and definitions of any terms that may not be clear.

5. PRINCIPLE OF INDIVIDUAL METHODS

5. A concise statement of the principle of the method is given to enable the user to decide whether the method is what he is seeking.

6. APPARATUS AND REAGENTS

6.1 The equipment and supplies required for the test are enumerated.

6.2 Test Solutions— Distilled water is to be used for making test solutions. The concentrations of baths are given in millilitres per litre (ml./l.) or grams per litre (g./l.). The qualities of chemicals used are given in each method. For crystalline substances, the amount of water of crystallisation is given, and for liquids, the density.

7. SPECIMEN

7.1 Directions are given for the preparation of the "specimen" or "composite specimen" to be tested and "test-control specimens" to be used.

7.2 By *specimen* is meant the small piece of material, usually taken from a larger sample or lot, that is to be submitted to the test.

7.3 By *composite specimen* is meant the specimen with attached undyed cloth used for evaluating staining.

7.4 By *test-control specimen* is meant a specimen of known behaviour used to ensure the carrying out of a test correctly.

7.5 Piece Goods— Specimens from woven and knit fabrics, felts, and other piece goods should be free from creases so that the treatment they receive may result in uniform action over the whole area.

7.6 Yarns— Yarns to be tested may be knit into fabric from which a specimen is taken. They may be wound parallel, for example on a U-shaped wire frame, and then sewn between the undyed cloths. For dry treatments, close winding on a card is recommended. For certain wet treatments without accompanying undyed material, hanks of yarn may be used, tied at both ends.

7.7 Fibres— Fibres may be tested by combing and compressing into a flat pad which may then be sewn between the white cloths.

7.8 Undyed Cloth for Staining—

7.8.1 The undyed cloth to be used for evaluating staining, if not otherwise specified, should be plain weave, medium weight, and free from finishes, residual chemicals, and chemically damaged fibres. Cotton and linen should be bleached; other materials should be cleaned to their usual degree of whiteness without bleaching.

7.8.2 The fluidity value of the cotton cloth used for staining may not exceed 5 rhes when measured in the standard cuprammonium solution by the method of Clibbens and Geake. Reference: *J. Textile Institute*, Vol. 19, page T 77 (1928).

8. CONDITIONING

8.1 Special conditioning of specimens usually is not necessary, but specimens, and the undyed cloths used with them, should be neither moist nor very dry. Tests in which differences in moisture content of the specimen and undyed cloths will influence the results should be in standard condition i.e. in moisture equilibrium with air having a temperature of 20°C. ±2°C. (68°F. ±4°F.) and relative humidity of 65% ±2%.

9. PROCEDURE

9.1 The series of operations through which the specimen is taken are described including the evaluation of colour change and staining of undyed cloth. The use of test-control specimens is called for in some of the methods to ensure the carrying out of the tests correctly.

9.2 Tolerances— Tolerances are given for numerical values of dimensions, temperatures, and times that are considered critical. If no tolerance is given, the precision of the measurements need be only that to be expected when using common instruments and reasonable care. The precision is further indicated by the number of significant figures in the values given.

9.3 Liquor Ratio— By *liquor ratio* is meant the ratio of the volume of the liquor used in the treatment expressed in millilitres to the weight of the specimen or composite specimen (specimen plus undyed material) expressed in grams.

9.4 Wetting out— Special care must be taken when wetting specimens that they are uniformly saturated. In particular, when wool or materials containing wool are to be wet out, it is necessary for them to be thoroughly kneaded by hand, with the flattened end of a glass rod, or with a mechanical device.

9.5 Spotting— For tests in which the material is spotted with water or a reagent and the drop worked in with a glass rod, care should be taken not to raise the surface of the material, otherwise there will be a change in the light reflection and hence in appearance.

9.6 Wetting to 100% Increase in Weight— When the material is to be wetted to contain its own weight of liquor, it may be saturated with the liquor and then drawn between two rubber rollers, squeezed by means of a rubber roller on a glass plate, or centrifuged. Wringing by hand does not give uniform wetting.

9.7 Temperature— Temperatures are given in degrees centigrade (°C.), usually with a tolerance of plus or minus 2 degrees (±2°C.). For reliable results, continuous temperature control is essential.

10. EVALUATION OF COLOUR FASTNESS

10.1 Colour fastness with respect to change in colour of the specimen in the test and to staining of undyed materials are evaluated separately. Specimens and undyed cloths should have cooled after drying and have regained their normal moisture content before the evaluation.

10.2 Colour Fastness with respect to Change in Colour— The change which occurs in a test may be a change in depth of colour, a change in hue, a change in brightness or any combination of these. Regardless of the character of the change, the evaluation is based upon the magnitude of the visual contrast between two coloured areas, one being the original material, the other the tested specimen. This contrast is compared visually with the contrast represented by the 5 pairs of neutral colour chips (or swatches of cloth) in the standard Grey Scale specified for the purpose in ISO/TC 38/SC 1/32 "Colour Fastness: Grey Scale for Evaluating Change in Colour". Each step of the scale bears a fastness number running from 5, no contrast, to 1, a large contrast. The fastness rating of the specimen is that number of the Grey Scale which corresponds to the contrast between the original and treated specimen. If the latter lies between

two of the controls of the Scale, the specimen is given an intermediate rating, e.g. 4–3, or 3–4, but intermediate ratings are unnecessary when the fastness is less than 3. A rating of 5 is given only when there is practically no difference between the tested specimen and the original material.

10.3 COLOUR FASTNESS WITH RESPECT TO STAINING— The degree of staining of the undyed cloth either by absorption of dye from the bath or by direct transfer of colour from the specimen is evaluated by visual inspection of the side of the undyed cloth which was in contact with the specimen. The colour of the bath is ignored. The standard Grey Scale for evaluating staining is used. It is specified in ISO/TC 38/SC 1/33, "Colour Fastness: Grey Scale for Evaluating Staining". The steps of this Scale run from 5, no contrast, represented by a pair of white chips (or swatches of cloth), to 1, a large contrast, represented by a white and a grey chip. The contrast between the original undyed cloth and that stained as a result of the test is rated with the aid of the Scale in a similar way to that described for change in colour in section 10.2. Staining is evaluated without reference to the depth of colour of the material tested. The staining is evaluated for each type of undyed material in the test.

10.4 INFLUENCE OF PRETREATMENTS AND AFTERTREATMENTS— It is well known that certain pretreatments and aftertreatments affect the fastness properties of coloured textiles. Thus any change in a coloured material that takes place as a result of applying a fastness test may not necessarily be due to lack of fastness in the dye itself.

11. CONDITIONS OF VIEWING AND ILLUMINATION IN EVALUATING COLOUR FASTNESS

11.1 In evaluating colour fastness, a piece of the original material and the tested specimen, or the undyed and stained cloths, are arranged side by side in the same plane and oriented in the same direction. Two or more layers are used if necessary to avoid effects of other backing on the appearance. The appropriate Grey Scale is placed nearby in the same plane. The surrounding field is uniform grey of a brightness slightly less than that of the darkest member of the Grey Scale for evaluating change in colour. The surfaces to be compared are illuminated by north sky light in the Northern Hemisphere, south sky light in the Southern Hemisphere, or equivalent source with illumination of 50 foot-candles or more. The light is incident upon the surfaces at approximately 45° and direction of viewing is approximately along the perpendicular to the plane of the surfaces.

12. REPORT

12.1 The information to be supplied in the report of the tests is indicated under this heading in the methods.

13. NOTES

13.1 Sources of supply of materials called for in the tests, references to literature, and other information not considered necessary for inclusion in the body of the test method, are given under "Notes".

ISO/TC 38/SC 1/32
Colour Fastness: Grey Scale for Evaluating Change in Colour

NOTE—This draft, based on ISO/TC 38/SC 1/7 and ISO/TC 38/SC 1/9, has been prepared as a result of the discussions of the Subcommittee in New York on 10–12th November 1952. Following the meetings the draft was circulated in preliminary form to the delegates for comment and criticism and this final form includes the suggestions which have been received. It is now before the Subcommittee for discussion at its next meeting.

1. PURPOSE AND SCOPE

1.1 This standard describes the Grey Scale for evaluating changes in colour of textiles in colour fastness tests and its use. A precise colorimetric specification of the Scale is given as a permanent record against which newly prepared working standards and standards that may have changed can be compared (See Section 4.1).

2. GREY SCALE FOR EVALUATING CHANGE IN COLOUR

2.1 THE SCALE consists of 5 pairs of grey colour chips (or swatches of grey cloth) each representing a visual difference in colour and contrast. The differences represented, which are determined by the Adams chromatic value formula, and corresponding numerical fastness ratings are as follows—

Colour Differences in N.B.S. Units (Judds)	Fastness Rating
0	5
1½	4
3	3
6	2
12	1

2.2 FASTNESS RATING 5 is represented on the scale by two identical chips (or swatches) mounted side by side, neutral grey in colour and having a reflectance of 12 ±1% (See Section 3). The colour difference is 0.

2.3 FASTNESS RATINGS 4 TO 1, INCLUSIVE, are represented by a "reference" chip (or swatch) identical with those used for Grade 5, paired with similar but lighter neutral grey chips (or swatches), the visual differences in the pairs being in geometric steps of colour difference, or contrast, as shown in the table.

2.4 USE OF THE SCALE— Place a part of the original textile and the tested specimen of it side by side in the same plane and oriented in the same direction. Place the Grey Scale nearby in the same plane. The surrounding field should be uniform grey of a brightness slightly less than that of the darkest member of the Grey Scale. If necessary to avoid effects of the backing on the appearance of the textiles, use two or more layers of the original textile under both original and tested specimen. Illuminate the surfaces with north sky light in the Northern Hemisphere, south sky light in the Southern Hemisphere, or equivalent source with illumination of 50 foot-candles or more. The light should be incident upon the surfaces at approximately 45° and the direction of viewing approximately perpendicular to the plane of the surfaces. Compare the visual difference between original and tested textile with the differences represented by the

Grey Scale. The fastness rating of the specimen is that number of the Grey Scale which corresponds to the contrast between the original and treated specimens. If the latter lies between two of the contrasts of the Scale, the specimen is given an intermediate assessment, e.g. 4–3 or 3–4, but intermediate classification is unnecessary when the fastness is less than 3. A rating of 5 is given only when there is no difference between the tested specimen and the original material.

2.5 Describing Colour Changes in Fastness Tests—

2.5.1 In using the Grey Scale as outlined in Section 2.4, the character of the change in colour whether in hue, depth, brightness, or any combination of these, is not rated, the overall difference, or contrast, between original and tested specimen is the basis for the evaluation.

2.5.2 If, as in rating dyestuffs on textiles, for example, it is desired to record the character of the change in colour of the textile in the test, appropriate qualitative terms may be added to the numerical rating, as illustrated by the following examples—

Rating	Meaning	
3	Contrast equal to Grade 3 of the Grey Scale	Change is a loss in depth of colour only
3 redder	Contrast equal to Grade 3 of the Grey Scale	No significant loss in depth, but colour has become redder
3 weaker, yellower	Contrast equal to Grade 3 of the Grey Scale	Loss in depth and change in hue have occurred
3 weaker, bluer, duller	Contrast equal to Grade 3 of the Grey Scale	Loss in depth and change in both hue and brightness have occurred
4–5 redder	Contrast intermediate between 4 and 5 of the Grey Scale	No significant loss in depth but colour has become slightly redder

2.5.3 When changes in colour occur in two or three directions it is considered neither feasible nor necessary to indicate the relative magnitude of each change, either in words (such as "much" or "little") or by number (e.g. 1 = small, 3 = large), or even by putting the larger change first (i.e. "bluer, duller" being different from "duller, bluer").

3. Colorimetric Specification of the Grey Scale

3.1 The precise colorimetric specification of the standard grey colours of the scale given in this section is derived from Section 2.

3.2 Actual Grey Scales used as working standards should approximate these colours closely, particularly in colour spacing.

3.3 The tristimulus values for the grey colours for 45° illuminating and normal viewing under C.I.E. standard source C are given in the following table.

Fastness Rating	Tristimulus Values for 45-normal illuminating and viewing under C.I.E. Standard Source C						N.B.S. units of Colour Difference Judd
	First Colour			Second Colour			
	X	*Y*	*Z*	*X*	*Y*	*Z*	
5	0·1176	0·1200	0·1417	0·1176	0·1200	0·1417	0·0
4	0·1176	0·1200	0·1417	0·1280	0·1306	0·1543	1·5
3	0·1176	0·1200	0·1417	0·1389	0·1417	0·1673	3·0
2	0·1176	0·1200	0·1417	0·1620	0·1652	0·1951	6·0
1	0·1176	0·1200	0·1417	0·2132	0·2175	0·2569	12·0

4. Notes and References

4.1 Grey Scales may be obtained from—

England, Society of Dyers and Colourists, 19 Piccadilly, Bradford, Yorkshire.

Germany, Deutsche Echtheitskommission, Krefeld, Adlerstrasse 18.

U.S.A., American Association of Textile Chemists and Colorists, Lowell Technological Institute, Lowell, Massachusetts.

ISO/TC 38/SC 1/33
Colour Fastness: Grey Scale for Evaluating Staining

Note—This draft, based on ISO/TC 38/SC 1/8, has been prepared as a result of the discussions of the Subcommittee in New York on 10–12th November 1952. Following the meetings the draft was circulated in preliminary form to the delegates for comments and criticism and this final form includes the suggestions which have been received. It is now before the Subcommittee for discussion at its next meeting.

1. Purpose and Scope

1.1 This standard describes the Grey Scale for evaluating staining of undyed textiles in colour fastness tests and its use. A precise colorimetric specification of the Scale is given as a permanent record against which newly prepared working standards and standards that may have changed can be prepared (See Section 4.1).

2. Grey Scale for Evaluating Staining

2.1 The Scale consists of 5 pairs of grey or white colour chips (or swatches of grey or white cloth) each representing a visual difference in colour or contrast. The differences represented, which are determined by the Adams chromatic value formula, and corresponding numerical fastness ratings are as follows—

Colour Differences in N.B.S. Units (Judds)	Fastness Rating
0	5
4	4
8	3
16	2
32	1

2.2 FASTNESS RATING 5 is represented on the scale by two identical chips (or swatches) mounted side by side, having a reflectance of not less than 85%. The colour difference is 0.

2.3 FASTNESS RATINGS 4 TO 1, INCLUSIVE, are represented by a reference chip (or swatch) identical with those used for rating 5, paired with similar but neutral grey chips (or swatches), the visual differences in the pairs being in geometric steps of colour difference, or contrast, as shown in the table.

2.4 USE OF THE SCALE— Place a piece of the unstained undyed textile and the stained piece of it side by side in the same plane and oriented in the same direction. Place the Grey Scale nearby in the same plane. The surrounding field should be uniform grey of a brightness slightly less than that of the darkest member of the Grey Scale for evaluating change in colour. If necessary to avoid effects of the backing on the appearance of the textiles, use two or more layers of the unstained undyed textile under both unstained and stained piece. Illuminate the surfaces with north sky light in the Northern Hemisphere, south sky light in the Southern Hemisphere, or equivalent source of illumination of 50 foot-candles or more. The light should be incident upon the surfaces at approximately 45° and the direction of viewing approximately perpendicular to the plane of the surfaces. Compare the visual difference between original undyed and stained pieces with the differences represented by the Grey Scale. The fastness rating of the specimen is that number of the Grey Scale which corresponds to the contrast between the original and treated specimens. If the latter lies between two of the contrasts of the Scale, the specimen is given an intermediate assessment, e.g. 4–3 or 3–4, but intermediate classification is unnecessary when the fastness is less than 3. A rating of 5 is given only there is no difference between the tested specimen and the original material.

3. COLORIMETRIC SPECIFICATION OF THE GREY SCALE

3.1 The precise colorimetric specification of the standard grey colours of the scale given in this section is derived from Section 2.

3.2 Actual Grey Scales used as working standards should approximate these colours closely, particularly in colour spacing.

3.3 The tristimulus values for the grey colours for 45° illuminating and normal viewing under C.I.E. standard source C are given in the following table.

Fastness Rating	Tristimulus Values for 45-normal illuminating and viewing under C.I.E. Standard Source C						N.B.S. Units of Colour Difference Judd
	First Colour			Second Colour			
	X	*Y*	*Z*	*X*	*Y*	*Z*	
5							
4							
3	[Data not yet available]						
2							
1							

4. NOTES AND REFERENCES

4.1 Grey Scales may be obtained from—

England, Society of Dyers and Colourists, 19 Piccadilly, Bradford, Yorkshire.

Germany, Deutsche Echtheitskommission, Krefeld, Adlerstrasse 18.

U.S.A., American Association of Textile Chemists and Colorists, Lowell Technological Institute, Lowell, Massachusetts.

ISO/TC 38/SC 1/34
Colour Fastness to Acid Spotting

NOTE—This draft, based on ISO/TC 38/SC 1/3, pages 14 and 15, has been prepared as a result of the discussions of the Subcommittee in New York on 10–12th November 1952. Following the meetings the draft was circulated in preliminary form to the delegates for comment and criticism and this final form includes the suggestions which have been received. It is now before the Subcommittee for discussion at its next meeting.

1. PURPOSE AND SCOPE

1.1 This method is intended for evaluating the resistance of the colour of textiles of all kinds in all forms to the action of dilute solutions of organic and mineral acids.

1.2 Three tests differing in severity are provided. Any or all may be used depending upon the nature of the fibre.

2. PRINCIPLE

2.1 Drops of the acid solution are worked into the textile with a glass rod and the changes in colour of the textile while it is still wet and after drying are assessed with a standard Grey Scale.

3. APPARATUS AND REAGENTS

3.1 Pipette or dropper

3.2 Glass rod with rounded end

3.3 Grey Scale for evaluating change in colour (See Section 7.1)

3.4 Acetic acid solution containing 300 g. glacial acetic acid per litre

3.5 Sulphuric acid solution containing 50 g. concentrated sulphuric acid (sp. gr. 1·84) per litre

3.6 Tartaric acid solution containing 100 g. tartaric acid per litre. (Especially for acetate fibre instead of acetic acid.)

4. SPECIMEN

4.1 If the textile to be tested is cloth, a piece at least 10 cm. × 4 cm. is required.

4.2 If the textile to be tested is yarn, knit it into cloth and use a piece at least 10 cm. × 4 cm. or from a number of lengths of it form a wick 10 cm. long and 0·5 cm. in diameter tied at both ends.

4.3 If the textile to be tested is bulk fibre, comb and compress enough of it to form a sheet at least 10 cm. × 4 cm.

5. Procedure

5.1 Spot the specimen with two drops of the appropriate solution at 25°C. ±2°C. (77°F. ±4°F.). Work the drops into the specimen with the glass rod.

5.2 Dry the specimen in air at 25°C. ±2°C. (77°F. ±4°F.).

5.3 Evaluate the change in colour with the Grey Scale (See Section 7.1).

6. Report

6.1 Report the numerical rating for change in colour for each kind of acid used (and for change in colour when wet if of interest).

7. Notes

7.1 See the following documents—

(*a*) Colour Fastness of Textiles: General Principles of Testing, ISO/TC 38/SC 1/31, *and*

(*b*) Colour Fastness: Grey Scale for Evaluating Change in Colour, ISO/TC 38/SC 1/32.

7.2 The change in colour of the specimen after it has been wet for 10 minutes may be observed. It is of interest for the evaluation of dyestuffs.

ISO/TC 38/SC 1/35

Colour Fastness to Alkali Spotting

Note—This draft, based on ISO/TC 38/SC 1/3, page 16, has been prepared as a result of the discussions of the Subcommittee in New York on 10–12th November 1952. Following the meetings the draft was circulated in preliminary form to the delegates for comment and criticism and this final form includes the suggestions which have been received. It is now before the Subcommittee for discussion at its next meeting.

1. Purpose and Scope

1.1 This method is intended for evaluating the resistance of the colour of textiles of all kinds in all forms to the action of dilute alkaline solutions.

2. Principle

2.1 Drops of a sodium carbonate solution are worked into the textile with a glass rod and the change in colour is evaluated with a standard Grey Scale.

3. Apparatus and Reagents

3.1 Pipette or dropper

3.2 Glass rod with rounded end

3.3 Grey Scale for evaluating change in colour (See Section 7.1)

3.4 Sodium carbonate solution containing 100 g. anhydrous sodium carbonate per litre.

4. Specimen

4.1 If the textile to be tested is cloth, a piece at least 10 cm. × 4 cm. is required.

4.2 If the textile to be tested is yarn, knit it into cloth and use a piece at least 10 cm. × 4 cm. or from a number of lengths of it form a wick 10 cm. long and 0·5 cm. in diameter tied at both ends.

4.3 If the textile to be tested is bulk fibre, comb and compress enough of it to form a sheet at least 10 cm. × 4 cm.

5. Procedure

5.1 Spot the specimen with two drops of the sodium carbonate solution at 25°c. ±2°c. (77°F. ±4°F.). Work the drops into the specimen with the glass rod. If penetration does not take place readily, immerse the specimen in the solution and squeeze so as to leave its own weight of solution in it. Dry the specimen in air at 25°c. ±2°c. (77°F. ±4°F.). Brush it to remove sodium carbonate residues.

5.2 Evaluate the change in colour with the Grey Scale (See Section 7.1).

6. Report

6.1 Report the numerical rating for change in colour.

7. Note

7.1 See the following documents—

(*a*) Colour Fastness of Textiles: General Principles of Testing, ISO/TC 38/SC 1/31.

(*b*) Colour Fastness: Grey Scale for Evaluating Change in Colour, ISO/TC 38/SC 1/32.

ISO/TC 38/SC 1/36

Colour Fastness to Bleaching, Hypochlorite

Note—This draft, based on ISO/TC 38/SC 1/5, has been prepared as a result of the discussions of the Subcommittee in New York on 10–12th November 1952. Following the meetings the draft was circulated in preliminary form to the delegates for comment and criticism and this final form includes the suggestions which have been received. It is now before the Subcommittee for discussion at its next meeting.

1. Purpose and Scope

1.1 This method is intended for evaluating the resistance of the colour of natural and regenerated cellulose textiles in all forms to the action of bleaching baths containing sodium and calcium hypochlorite in concentrations normally used in commercial bleaching.

2. Principle

2.1 A specimen of the textile is agitated in a sodium hypochlorite solution, rinsed in water, agitated in sodium bisulphite solution, rinsed, and dried. The change in colour is evaluated with a standard Grey Scale.

3. Apparatus and Reagents

3.1 Sodium hypochlorite solution containing 2 g. of available chlorine per litre, buffered at pH 11·0 ±0·2 with 10 g. anhydrous sodium carbonate per litre, temperature 25°c. ±2°c. (77°F. ±4°F.). Weight of solution used to be 50 times that of the specimen. Sodium hypochlorite of the following composition shall be used—

Sodium hypochlorite	NaOCl	14% to 16%
Sodium chloride	NaCl	12% to 17%
Sodium hydroxide	NaOH	2·0% maximum
Sodium carbonate	Na_2CO_3	2·0 %maximum
Iron	Fe	0·001% maximum

3.2 Sodium bisulphite solution containing 5 g. $NaHSO_3$ per litre. Weight of solution used to be 50 times the weight of the specimen.

3.3 Container for specimen and bleaching solution that can be closed (See Section 5.1).

3.4 Grey Scale for evaluating change in colour.

TABLE I

Composition of Bleach Bath and Conditions of Use

Starting Bath (per litre of distilled water)	Bath 1 For Natural and Regenerated Cellulose	Bath 2 For Natural and Regenerated Cellulose	Bath 3 For Wool and Acetate	Bath 4 For Silk
Hydrogen peroxide (*a*), ml.	5	—	20	20
Sodium peroxide (*b*), g.	—	3	—	—
Sodium silicate (*c*), ml.	5	—	—	5
Sodium pyrophosphate (*d*), g.	—	—	5	—
Magnesium chloride (*e*), g.	0·1	—	—	0·1
pH, initial value ±0·2 (*f*)	10·5	11·5	9·3	10·0
Temperature, °C. ±2°	90	80	50	70
Temperature, °F. ±4°	194	176	122	158
Duration of treatment, hours	1	1	2	2
Liquor ratio, vol. of liquor in ml : weight of cloth in g.	30 : 1	30 : 1	30 : 1	30 : 1

(*a*) 30% H_2O_2 by volume
(*b*) 100% Na_2O_2
(*c*) About 26% SiO_2 and 10% Na_2O, *d* 1·32, 35° Bé
(*d*) $Na_4P_2O_7,10H_2O$
(*e*) $MgCl_2$
(*f*) Adjust by addition of NaOH solution, if necessary

4. SPECIMEN

4.1 If the textile to be tested is fabric, use a specimen at least 10 cm. × 4 cm. (4 inches × 1·5 inches).

4.2 If the textile to be tested is yarn, knit it into a fabric and use a specimen of it at least 10 cm. × 4 cm. or make a wick of parallel lengths 10 cm. (4 inches) long and about 0·5 cm. ($\frac{3}{16}$ inch) in diameter tied near both ends.

4.3 If the textile to be tested is fibre, comb and compress some of it into a sheet at least 10 cm. × 4 cm. and sew the sheet on a piece of unbleached, desized cotton cloth to support the fibres.

5. PROCEDURE

5.1 Wet out the specimen in distilled water, remove the excess water, and place the specimen in an open or loosely rolled form in the sodium hypochlorite solution (See Section 3.1). Close the container and keep the specimen in the solution at 25°C. ±2°C (77°F. ±4°F.) for 60 minutes. Avoid exposure to direct sunlight.

5.2 Wash the specimen thoroughly in cold running water and then stir it for 10 minutes in the sodium bisulphite solution at 25°C. ±2°C. (77°F. ±4°F.).

5.3 Wash the specimen thoroughly in cold running water, remove the excess water, and dry the specimen in air at a maximum temperature of 60°C. (140°F.).

5.4 Evaluate the change in colour of the specimen with the Grey Scale (See Section 7.1).

6. REPORT

6.1 Report the numerical rating for change in colour.

7. NOTES

7.1 See the following documents—

(*a*) Colour Fastness of Textiles: General Principles of testing, ISO/TC 38/SC 1/31.
(*b*) Colour Fastness: Grey Scale for Evaluating Change in Colour: ISO/TC 38/SC 1/32.

ISO/TC 38/SC 1/37

Colour Fastness to Bleaching, Peroxide

NOTE—This draft, based on ISO/TC 38/SC 1/15, pages 3 and 4, has been prepared as a result of the discussions of the Subcommittee in New York on 10–12th November 1952. Following the meetings the draft was circulated in preliminary form to the delegates for comment and criticism and this final form includes the suggestions which have been received. It is now before the Subcommittee for discussion at its next meeting.

1. PURPOSE AND SCOPE

1.1 This method is intended for evaluating the resistance of the colour of textiles of all kinds except polyamide, in all forms, to the action of bleach baths containing peroxide at concentrations commonly employed in textile processing.

2. PRINCIPLE

2.1 A specimen of the textile in contact with specified undyed cloths is immersed in the bleaching solution, rinsed, dried, and the change in colour and staining are evaluated with standard Grey Scales.

3. APPARATUS AND REAGENTS

3.1 Test tube, diameter and length such that the test specimen roll will have a reasonably snug fit in the tube and be covered by the bleach solution.

3.2 Reflux condenser, fitting the test tube, may be used to reduce evaporation from the bleach bath during the test.

3.3 Bleach bath of the composition given in Table I.

3.4 Two undyed cloths, each 10 cm. × 4 cm., one piece made of the same kind of fibre as that in the textile to be tested, the second piece made of the fibre indicated in Table II, or as otherwise specified (See Section 7.1).

TABLE II

If First Piece is—	Second Piece to be—
Wool, silk, linen, viscose rayon	Cotton
Cotton, acetate	Viscose rayon

3.5 Grey Scales for evaluating the change in colour and the staining of the white cloths (See Section 7.2).

4. SPECIMEN

4.1 If the textile to be tested is cloth, place a 10 cm. × 4 cm. (about 4 × 1½ inches) specimen of it between the two pieces of undyed cloth (See Section 3.3), and sew along all four sides to form a composite specimen.

4.2 If the textile to be tested is yarn, knit it into cloth and treat it as in 4.1, or form a layer of parallel lengths of it between two undyed cloths, and sew around the four sides to hold the yarns in place.

4.3 If the textile to be tested is fibre, comb and compress some of it into a sheet 10 cm. × 4 cm. in size, place the sheet between the two undyed cloths, and sew around the four sides to form a composite specimen.

5. PROCEDURE

5.1 Loosely roll the composite specimen in the direction of the long edge, place the 4 cm. roll in the test tube, in the appropriate bleach solution (Table I), and keep it well covered for the time and at the temperature indicated in the table.

5.2 Remove the specimen, rinse it for 10 minutes in flowing cold tap water and squeeze it. Remove the stitching on two long sides and one short side, open it out, and dry at a temperature not exceeding 60°C. (140°F.) with the three parts in contact only at the one line of stitching.

5.3 Evaluate the change in colour of the specimen and the staining of the undyed cloths with the appropriate standard Grey Scales (See Section 7.2).

6. REPORT

6.1 Report the bleach solution used, the numerical rating for colour change, and the numerical rating for staining of each kind of undyed fibre tested.

7. NOTES

7.1 The undyed cloths should be plain weave, medium weight, and free from finishes, residual chemicals, and chemically damaged fibres. Cotton and linen should be bleached; other materials should be cleaned to their usual degree of whiteness without bleaching.

7.2 See the following documents—

(*a*) Colour Fastness of Textiles: General Principles of Testing, ISO/TC 38/SC 1/31.

(*b*) Colour Fastness: Grey Scale for Evaluating Change in Colour, ISO/TC 38/SC 1/32.

(*c*) Colour Fastness: Grey Scale for Evaluating Staining, ISO/TC 38/SC 1/33.

ISO/TC 38/SC 1/38

Colour Fastness to Carbonising with Aluminium Chloride

NOTE—This draft, based on ISO/TC 38/SC 1/3, page 17, and AFNOR Standard NF G 07–019 accompanying ISO/TC 38/SC 1/20, has been prepared as a result of the discussions of the Subcommittee in New York on 10–12th November 1952. Following the meetings the draft was circulated in preliminary form to the delegates for comment and criticism and this final form includes the suggestions which have been received. It is now before the Subcommittee for discussion at its next meeting.

1. PURPOSE AND SCOPE

1.1 This method is intended for evaluating the resistance of the colour of wool and part wool textiles to the manufacturing operation designed to remove vegetable impurities by treatment with aluminium chloride at high temperatures. In particular, it is used for wool textiles containing acetate or polyamide fibres.

2. PRINCIPLE

2.1 A specimen containing the aluminium chloride solution is dried and baked, rinsed and neutralised, and the changes in colour after rinsing, neutralising, and drying are evaluated with a standard Grey Scale.

3. APPARATUS AND REAGENTS

3.1 Oven for drying specimens in air at 60°C. ±2°C. (140°F. ±4°F.) and baking in air at 115°C. ±2°C. (239°F. ±4°F.).

3.2 Aluminium chloride solution (sp. gr. 1·037) containing 51·4 g. Al_2Cl_6 per litre.

3.3 Ammonium hydroxide solution containing 2 ml. of 20% NH_4OH per litre.

3.4 Grey Scale for assessing change in colour (See Section 7.1).

4. SPECIMEN

4.1 If the textile to be tested is cloth, a piece at least 10 cm. × 4 cm. is required.

4.2 If the textile to be tested is yarn, knit it into cloth and use a piece at least 10 cm. × 4 cm. or from a number of lengths of it form a wick 10 cm. long and 0·5 cm. in diameter tied at both ends.

4.3 If the textile to be tested is bulk fibre, comb and compress enough of it to form a sheet at least 10 cm. × 4 cm.

5. PROCEDURE

5.1 Immerse the specimen in the aluminium chloride solution for 15 minutes at 25°C. ±2°C. (77°F. ±4°F.) liquor ratio 20 : 1. Squeeze it to leave in 80% of its weight of solution.

5.2 Dry the specimen by hanging it in an oven for 30 minutes, or longer if necessary, at 60°C ±2°C. (140°F. ±4°F.). Then bake it by heating for 15 minutes at 115°C. ±2°C. (239°F. ±2°F.).

5.3 Rinse the specimen for 5 minutes in cold running water and then divide it into two equal parts. Dry one half in air at a temperature not exceeding 60°C. (140°F.).

5.4 Agitate the other half for 30 minutes at 25°C. ±2°C. (77°F. ±4°F.) in the ammonium hydroxide solution, liquor ratio 40 : 1. Then rinse it for 5 minutes in cold water running and dry it in air at a temperature not exceeding 60°C. (140°F.).

5.5 Assess the change in colour of each half of the specimen with the Grey Scale (See Section 7.1).

6. REPORT

6.1 Report the numerical rating for change in colour for both the rinsed and the neutralised portions of the specimen.

7. NOTES

7.1 See the following documents—

(*a*) Colour Fastness of Textiles: General Principles of Testing, ISO/TC 38/SC 1/31.

(*b*) Colour Fastness: Grey Scale for Evaluating Change in Colour, ISO/TC 38/SC1/32.

ISO/TC 38/SC 1/39

Colour Fastness to Carbonising with Sulphuric Acid

NOTE—This draft, based on ISO/TC 38/SC 1/3, page 17, has been prepared as a result of the discussions of the Subcommittee in New York on 10–12th November 1952. Following the meetings the draft was circulated in preliminary form to the delegates for comment and criticism and this final form includes the suggestions which have been received. It is now before the Subcommittee for discussion at its next meeting.

1. PURPOSE AND SCOPE

1.1 This method is intended for evaluating the resistance of the colour of wool and part wool textiles to the manufacturing operation designed to remove vegetable impurities by treatment with sulphuric acid at high temperatures.

2. PRINCIPLE

2.1 A specimen containing the sulphuric acid solution is dried and baked, rinsed and neutralised, and the changes in colour after rinsing, neutralising, and drying are evaluated with a standard Grey Scale.

3. APPARATUS AND REAGENTS

3.1 Oven for drying specimens in air at 60°C. ±2°C. (140°F. ±4°F.) and baking in air at 105°C. ±2°C. (221°F. ±4°F.).

3.2 Sulphuric acid solution containing 50 g. concentrated sulphuric acid (sp. gr. 1·84) per litre.

3.3 Sodium carbonate solution containing 2 g. anhydrous sodium carbonate per litre.

3.4 Test-control [to be inserted] (See Section 7.1).

3.5 Grey Scale for assessing change in colour (See Section 7.2).

4. SPECIMEN

4.1 If the textile to be tested is cloth, a piece at least 10 cm. × 4 cm. is required.

4.2 If the textile to be tested is yarn, knit it into cloth and use a piece at least 10 cm. × 4 cm. or from a number of lengths of it form a wick 10 cm. long and 0·5 cm. in diameter tied at both ends.

4.3 If the textile to be tested is bulk fibre, comb and compress enough of it to form a sheet at least 10 cm. × 4 cm.

5. PROCEDURE

5.1 Carry out the operations described in Section 5.2 to 5.7 inclusive with the composite specimen and the composite test-control specimen in parallel, in separate baths.

5.2 Immerse the specimen in the sulphuric acid solution for 15 minutes at 25°C. ±2°C. (77°F. ±4°F.) liquor ratio 20 : 1. Squeeze it to leave in 80% of its weight of solution.

5.3 Dry the specimen by hanging it in an oven for 30 minutes or longer if necessary, at 60°C. ±2°C. (140°F. ±4°F.). Then bake it by heating for 15 minutes at 105°C. ±2°C. (221°F. ±4°F.).

5.4 Rinse the specimen for 5 minutes in cold, running water and then divide it into two equal parts. Dry one half in air at a temperature not exceeding 60°C. (140°F.).

5.5 Agitate the other half for 30 minutes at 25°C. ±2°C. (77°F. ±4°F.) in the sodium carbonate solution, liquor ratio 40 : 1. Then rinse it for 5 minutes in cold running water and dry it in air at a temperature not exceeding 60°C. (140°F.).

5.6 Evaluate the effect on the test-control with the Grey Scale. If the results obtained differ significantly from the following—

[to be supplied]

the test has not been carried out correctly and the operations described in Section 5.1 to 5.7 inclusive must be repeated with fresh composite specimens and a fresh composite test-control specimen.

5.7 Assess the change in colour of each half of the specimen with the Grey Scale (See Section 7.2).

6. REPORT

6.1 Report the numerical rating for change in colour for both the rinsed and the neutralised portions of the specimen.

7. NOTES

7.1 [To be supplied.]

7.2 See the following documents—

(*a*) Colour Fastness of Textiles: General Principles of Testing, ISO/TC 38/SC 1/31.

(*b*) Colour Fastness: Grey Scale for Evaluating Change in Colour, ISO/TC 38/SC 1/32.

ISO/TC 38/SC 1/40

Colour Fastness to Chlorination, Acid

NOTE—This draft, based on ISO/TC 38/SC 1/4, dated 1st September 1952, pages 1 and 2 has been prepared as a result of the discussions of the Subcommittee in New York on 10–12th November 1952. Following the meetings the draft was circulated in preliminary form to the delegates for comment and criticism and this final form includes the suggestions which have been received. It is now before the Subcommittee for discussion at its next meeting.

1. PURPOSE AND SCOPE

1.1 This method is intended for evaluating the resistance of the colour of textiles of all kinds in all forms to the manufacturing operation in which an acid hypochlorite solution is used to prevent wool textiles from shrinking.

2. PRINCIPLE

2.1 A specimen of the textile in contact with undyed textiles of interest is treated successively with solutions of hydrochloric acid, sodium hypochlorite, and sodium sulphite, rinsed and dried. The change in colour of the specimen and staining of the undyed textiles are evaluated with standard Grey Scales. A test control fabric is used.

3. APPARATUS AND REAGENTS

3.1 Yarns of scoured unbleached undyed wool, undyed bleached cotton, and other fibres as desired for evaluation of staining if fabrics or yarns are to be tested; comparable undyed fabrics if fibres are to be tested.

3.2 Hydrochloric acid solution containing 6 ml. hydrochloric acid (sp. gr. 1·16) per litre.

3.3 Sodium hypochlorite solution containing 2 g. of available chlorine per litre. Sodium hypochlorite of the following composition shall be used—

Sodium hypochlorite	NaOCl	14% to 16%
Sodium chloride	NaCl	12% to 17%
Sodium hydroxide	NaOH	2·0% maximum
Sodium carbonate	Na_2CO_3	2·0% maximum
Iron	Fe	0·001% maximum

3.4 Sodium sulphite solution containing 3 g. $Na_2SO_3.7\,H_2O$ per litre.

3.5 Test-control dyeing [to be inserted] (See Section 7.1).

3.6 Standard Grey Scales (See Section 7.2).

4. SPECIMEN

4.1 If the textile to be tested is cloth, sew stitches of the undyed yarns (See Section 3.1) at intervals of approximately 1 cm. (0·4 inch) into a specimen of the cloth at least 7·5 cm. × 5 cm. (3 inches × 2 inches). The combined weight of the undyed yarns should be approximately one-third of that of the cloth.

4.2 If the textile to be tested is yarn, knit it into fabric and make a composite specimen from it as in Section 4.1.

4.3 If the textile to be tested is fibre, comb and compress some of it into a sheet at least 7·5 cm. × 5 cm. (3 inches × 2 inches) in size, place the sheet between the undyed wool and undyed cotton or other fabrics, and sew the three together with stitching at intervals of 1 cm. (0·4 inch). The weight of the coloured textile should approximate that of the undyed wool.

4.4 Test control.—Prepare a composite specimen of the test control dyeing (See Section 3.5) in the way outlined for cloth in Section 4.1).

5. PROCEDURE

5.1 Carry out the operations described in Sections 5.2 to 5.5 inclusive with the composite specimens and the composite test-control specimen in parallel in separate baths.

5.2 Immerse the composite specimen in the hydrochloric acid solution, liquor ratio 12·5 : 1 for 10 minutes at 25°C. ±2°C. (77°F. ±4°F.).

5.3 Add an equal volume of the sodium hypochlorite solution (See Section 3.3) and keep the composition specimen immersed for a further 10 minutes.

5.4 Rinse the composite specimen thoroughly in water and then immerse it in the sodium sulphite solution (See Section 3.4) for 10 minutes at 35°C. to 40°C. (95°F. to 104°F.).

5.5 Thoroughly rinse the composite specimen from Section 5.4 in water and dry it in air at a temperature not exceeding 60°C. (140°F.).

5.6 [Interpretation of results with the test-control specimen to be inserted.]

5.7 Evaluate the change in colour of the specimen and the staining of the undyed materials with the Grey Scales (See Section 7.2).

6. REPORT

6.1 Report the numerical rating for change in colour and the numerical rating for staining of each kind of undyed fibre tested.

7. NOTES

7.1 [Information about the test-control dyeing to be provided.]

7.2 See the following documents—

(*a*) Colour Fastness of Textiles: General Principles of Testing, ISO/TC 38/SC 1/31.

(*b*) Colour Fastness: Grey Scale for Evaluating Change in Colour, ISO/TC 38/SC 1/32.

(*c*) Colour Fastness: Grey Scale for Evaluating Staining, ISO/TC 38/SC 1/33.

ISO/TC 38/SC 1/41

Colour Fastness to Light: Daylight

Note—This draft, based on ISO/TC 38/SC 1/6, ISO/TC 38/SC 1/16, ISO/TC 38/SC 1/20, has been prepared as a result of the discussions of the Subcommittee in New York on 10–12th November 1952. Following the meetings the draft was circulated in preliminary form to the delegates for comment and criticism and this final form includes the suggestions which have been received. It is now before the Subcommittee for discussion at its next meeting.

1. PURPOSE AND SCOPE

1.1 This method is intended for evaluating the resistance of the colour of textiles of all kinds and in all forms to the action of daylight (See Section 7.1).

1.2 The method may be used for evaluating dyestuffs by applying the dyestuff to textiles under specified conditions and depth of shade and testing the dyed textiles.

2. PRINCIPLE

2.1 A specimen of the textile is exposed to daylight under prescribed conditions, including protection from rain, along with eight dyed wool standards. The fastness is evaluated by comparing the fading of the textile with that of the standards.

3. STANDARDS AND EQUIPMENT

3.1 Standards

3.1.1 The standards for this test are blue dyed wool cloths made with the dyestuffs listed below. They range from No. 1, very low lightfastness, to No. 8, very high lightfastness (See Section 7.2).

Fastness Rating	Dyestuff
1	Acilan [?] Brilliant Blue FFR (Bayer)
2	Acilan [?] Brilliant Blue FFB (Bayer)
3	Supranoleyanin 6B (Bayer) *or* Brilliantcyanin 6B (Ciba)
4	Polar Blue G Conc. (Geigy)
5	Solway Blue RS (I.C.I.) *or* Acilan Fast Blue RX (Bayer) *or* Alizarine Light Blue R (Sandoz)
6	Acid Alizarine Sapphire 2JL (C.F.M.C.) *or* Alizarine Light Blue 4GL (Sandoz)
7	Soledon Blue 4BC powder (I.C.I.)
8	Indigosol Blue AGG (Durand) *or* Anthrasol Blue AGG (Hœchst)

3.1.2 Master sets of the standard dyeings are to be filed in each country for reference.

3.2 Exposure Rack, facing due south in the Northern Hemisphere, north in the Southern Hemisphere and sloping at an angle of 45°, in a location such that shadows of surrounding objects will not fall on the exposed textiles, covered with

window glass to protect the specimens from the weather, and provided with adequate ventilation.

3.3 Opaque cardboard or other thin opaque material, for example, thin sheet aluminium, or cardboard covered with aluminium foil.

3.4 Grey Scale for evaluating change in colour (See Section 7·3).

4. SPECIMEN

4.1 An area of the textile not less than 1 cm. × 6 cm. ($\frac{3}{8}$ inch × $2\frac{3}{8}$ inches) is used when the several periods of exposure are made side by side on the same piece, which is the preferred practice. The specimen may be: A strip of cloth, yarns wound close together on a card or laid parallel and fastened on a card, or a mat of fibres combed and compressed to give a uniform surface and fastened on a card.

4.2 To facilitate handling the specimen or specimens to be tested and similar strips of the standards, may be mounted on a card in some such arrangement as indicated in Section 7.5.

5. PROCEDURE

5.1 Expose the specimen, or a group of specimens, and the standards simultaneously to daylight under the conditions enumerated in Section 3.2 in such a manner and for such times as are necessary to fully evaluate the light fastness of each specimen relative to that of the standards. (See Section 7.5 for two suggested methods of procedure.)

5.2 PRELIMINARY EVALUATION OF LIGHT FASTNESS— When a change in colour (See Section 7.6) of the specimen under test can just be perceived, note the number of the standard showing a similar change or, alternatively, when a change in colour of standard 3 can just be perceived, evaluate any change that has occurred in the specimen in comparison with the changes in standards 1, 2, and 3 (See Section 7.4). Continue to expose after the preliminary evaluation as required to evaluate fully light fastness.

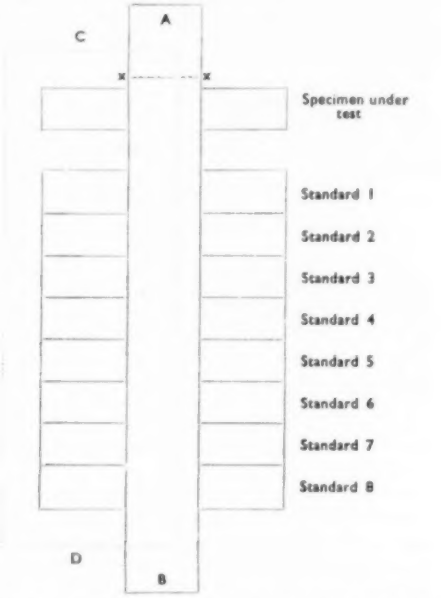
5.3 EVALUATION OF LIGHT FASTNESS—Compare the changes in colour of the test specimen with the changes which have occurred in the standards (See Section 7.6). The light fastness of the specimen is the number of the standard which shows similar changes in colour (visual contrast between exposed and unexposed parts in the test). If the specimen shows changes in colour intermediate between two standards, an appropriate half-rating may be given e.g. light fastness 3–4 means the textile is not as fast as standard 4, but faster than standard 3.

5.4 If the colour of the specimen is more fugitive than that of standard No. 1, a rating of 1 is given.

5.5 If the light fastness is 4 or higher, the evaluation based on the first perceptible change (See Section 5.2) becomes significant and if this preliminary evaluation is 3 or lower, it is to be included in the rating in brackets. For example, a rating of 6[3] indicates that the specimen changed very slightly in the test when standard 3 just began to fade, but that on continuing the exposure the resistance to light was equal that of standard 6.

6. REPORT

6.1 Report the numerical rating for light fastness and if the preliminary evaluation was 3 or lower report this figure in brackets.



AB Opaque cover. May be hinged at *zz* so that it can be lifted and returned to the same place over the specimen and standards.
CD Second cover.

FIG. 1— Mounting for Method 1

7. NOTES

7.1 The use of fading lamps rather than daylight is common practice and the results obtained with such tests are undoubtedly useful where speed is essential. The results obtained for many textiles differ, however, from the results obtained with daylight exposure.

7.2 Sets of the light fastness standards can be obtained from the British Standards Institution, British Standards House, 2 Park Street, London, W.1. They are specially dyed to match a Master set of standards in colour and in fading behaviour. It has been found when repeated dyeings of the dyed standards are made that the amount of dyestuff required to match the previous lot is often different from that originally used. The dyeing strengths would, therefore, be misleading and they are intentionally omitted from this listing.

7.3 Colour Fastness of Textiles: General Principles of Testing, ISO/TC 38/SC 1/31, and Colour Fastness: Grey Scale for Evaluating Change in Colour, ISO/TC 38/SC 1/32.

7.4 Phototropic specimens must be allowed to condition in the dark at room temperature for 2 hours before assessing the change in colour.

7.5 Suggested procedures for mounting and testing for light fastness:

7.5.1 METHOD 1—

7.5.1.1 This method is considered ideal and should be used in cases of dispute. It requires one set of standards for each specimen under test and is therefore impracticable when a large number of

specimens have to be tested concurrently; in such cases, Method 2 (See Section 7.5.2) is suggested.

7.5.1.2 Arrange the specimen to be tested and the standards as shown in Fig. 1 with an opaque cover across the middle third of the specimen and standards. Expose to daylight under the conditions enumerated in Section 3.2. Follow the effect of light by removing the cover and inspecting the specimen frequently. When a change can just be perceived, note the number of the standard showing a similar change.

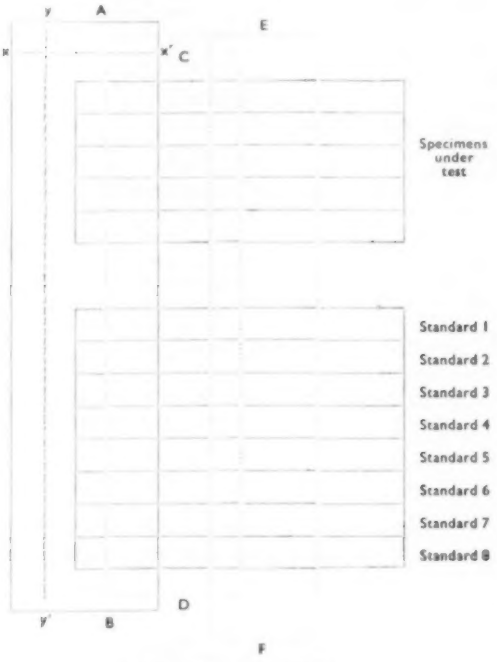


Fig. 2—Mounting for Method 2

7.5.1.3 Continue to expose until the contrast between the exposed and covered portion of the specimen is equal to the contrast between the two grey colour chips illustrating Grade 3 on the Grey Scale (See Section 7.3). Cover a second one-third of the specimen and standards with an additional opaque cover, *CD* in Fig. 1.

7.5.1.4 Continue to expose until the contrast between the fully exposed and the unexposed portion is equal to Grade 2 on the Grey Scale. If standard 7 fades to a contrast equal to Grade 3 on the Grey Scale before the specimen does, the exposure may be concluded at this stage. (When a specimen has a light fastness of 7 or over, it would require unduly long exposure to produce a contrast equal to Grade 2; moreover this contrast would be impossible to obtain when the light fastness is 8. Evaluations in the region 7 to 8 are, therefore, made when the contrast produced on standard 7 is equal to Grade 3, the time required to produce this contrast being long enough to eliminate any error which might result from inadequate exposure.)

7.5.1.5 Evaluation of light fastness. The specimen now shows three separate areas—an unexposed region and two areas which have been changed to different degrees. Compare these changes with the changes which have occurred in the standards and rate light fastness as in Section 5.3.

7.5.2 Method 2—

7.5.2.1 This method should be used when a large number of specimens have to be tested concurrently. It enables a number of specimens differing in light fastness to be rated against a single set of standards.

7.5.2.2 Arrange the specimens to be tested and the standards as shown in Fig. 2, with card *AB* covering one quarter of the total length of each specimen and standard. Expose to daylight under the conditions enumerated in Section 3.2. Follow the effect of light by lifting the cover periodically and inspecting the *standards*. When a change in standard 3 can just be perceived, inspect the specimens and rate their light fastness by comparing any change that has occurred with the changes that have occurred in standards 1, 2, and 3.

7.5.2.3 Replace the card in exactly the same position and continue to expose until a change in standard 4 can just be perceived; at this point fix an additional cover, *CD*, in the position shown in Fig. 2, overlapping the first cover.

7.5.2.4 Continue to expose until a change in standard 6 can just be perceived; then fix the final cover, *EF*, in the position shown, the other two covers remaining in position. Expose until either: (*a*) a contrast is produced on standard 7 equal to the contrast illustrating Grade 3 of the Grey Scale, or (*b*) a contrast equal to Grade 2 has been produced on the most resistant specimen, whichever occurs first. Remove the three covers.

7.5.2.5 Compare the changes in the specimens with those in the standards and rate light fastness as in Section 5.3.

7.6 The term "change in colour" includes not only true "fading", i.e. destruction of dyestuff, but also changes in hue, depth, brightness, or any combination of these characteristics of colour.

ISO/TC 38/SC 1/42

Colour Fastness to Mercerising

Note—This draft, based on ISO/TC 38/SC 1/3, page 20, has been prepared as a result of the discussions of the Subcommittee in New York on 10–12th November 1952. Following the meetings the draft was circulated in preliminary form to the delegates for comment and criticism and this final form includes the suggestions which have been received. It is now before the Subcommittee for discussion at its next meeting.

1. Purpose and Scope

1.1 This method is intended for evaluating the resistance of the colour of cotton yarns and mixed fibre yarns containing other natural cellulosic fibres with cotton to the action of concentrated solutions of sodium hydroxide used in mercerising.

2. Principle

2.1 A specimen of the textile in contact with specified undyed cotton cloth is treated with the sodium hydroxide solution rinsed, acidified, rinsed and dried. The change in colour of the specimen and staining of the undyed cloth are evaluated with standard Grey Scales.

3. APPARATUS AND REAGENTS

3.1 Undyed, bleached, cotton cloth at least 10 cm. × 10 cm. for evaluating staining.

3.2 Frame for holding specimen (See Section 4).

3.3 Sodium hydroxide solution, 300 g. sodium hydroxide per litre.

3.4 Sulphuric acid solution, 5 ml. concentrated sulphuric acid (sp. gr. 1·84) per litre.

3.5 Standard Grey Scales (See Section 7.1).

4. SPECIMEN

4.1 If the textile to be tested is cloth, sew a specimen of it at least 10 cm. × 10 cm. to an equal sized piece of the undyed cloth, Section 3.1, along all four sides. Fasten this composite specimen firmly, but without excessive tension, to a frame with the coloured material uppermost.

4.2 If the textile to be tested is yarn or thread wind it firmly, but without excessive tension, on a rigid frame with the strands close together and parallel to provide an area at least 10 cm. × 10 cm. Sew an equal sized piece of the undyed cloth to this area along the two sides across the strands, with the coloured material uppermost.

5. PROCEDURE

5.1 Immerse the composite specimen in the sodium hydroxide solution at 20°C. ±2°C. (68°F. ±4°F.) for 5 minutes. Rinse it in water at 70°C. ±2°C. (158°F. ±4°F.) for 5 minutes and in cold running water for 5 minutes.

5.2 Remove it from the frame and immerse it in the sulphuric acid solution for 5 minutes to acidify it. Wash it in cold running water until neutral.

5.3 Remove the stitching along three sides (one side for yarns and threads) and dry in air at a temperature not exceeding 60°C. (140°F.) taking care that the undyed cloth and coloured material are kept apart except at the stitching.

5.4 Evaluate the change in colour of the specimen and the staining of the undyed cotton cloth with the Grey Scales (See Section 7.1).

6. REPORT

6.1 Report the numerical rating for change in colour and the numerical rating for staining of undyed cotton cloth.

7. NOTE

7.1 See the following documents—

(*a*) Colour Fastness of Textiles: General Principles of Testing, ISO/TC 38/SC 1/31.

(*b*) Colour Fastness: Grey Scale for Evaluating Change in Colour, ISO/TC 38/SC 1/32.

(*c*) Colour Fastness: Grey Scale for Evaluating Staining, ISO/TC 38/SC 1/33.

ISO/TC 38/SC 1/43

Colour Fastness to Metals in the Dyebath: Chromium Salts

NOTE—This draft, based on ISO/TC 38/SC 1/4, page 3, has been prepared as a result of discussions of the Subcommittee in New York, November 1952.

1. PURPOSE AND SCOPE

1.1 This method is intended for evaluating the effect on the shade of a dyestuff when dyed in the presence of hexavalent chromium salts.

2. PRINCIPLE

2.1 The difference in shade between dyeings made with and without potassium dichromate is evaluated with the standard Grey Scale.

3. APPARATUS AND REAGENTS

3.1 Two pieces of undyed light wool serge of a size suitable for laboratory dyeing.

3.2 Two dye baths and solutions normal for the dyestuff.

3.3 Potassium dichromate solution containing 1% $K_2Cr_2O_7$ on the weight of one piece of the wool (See Section 3.1).

3.4 Grey Scale for evaluating change in colour (See Section 7.1).

4. SPECIMEN

4.1 (See Section 3.1).

5. PROCEDURE

5.1 Make two dyeings of the dye at the standard depth of shade on the wool using the normal method for the dyestuff under examination and a liquor ratio of 40 : 1.

5.2 On completion of the dyeing add to one of the dyebaths sufficient potassium dichromate solution to give 1% $K_2Cr_2O_7$ on the weight of the wool. Maintain the bath at the boil for a further 60 minutes.

5.3 Compare the shade of the dyeing in the presence of potassium dichromate with that of the dyeing without and evaluating the difference with the Grey Scale.

6. REPORT

6.1 Report the dyestuff used, the method and strength of dyeing, and the numerical rating of the change in shade.

7. NOTES

7.1 See the following documents—

(*a*) Colour Fastness of Textiles: General Principles of Testing, ISO/TC 38/SC 1/31.

(*b*) Colour Fastness: Grey Scale for Evaluating Change in Colour, ISO/TC 38/SC 1/32.

ISO/TC 38/SC 1/44

Colour Fastness to Metals in the Dyebath: Iron and Copper

NOTE—This draft, based on ISO/TC 38/SC 1/3, pages 3 and 4, 1st September 1952, has been prepared as a result of discussions of the Subcommittee in New York, November 1952.

1. PURPOSE AND SCOPE

1.1 This method is intended for evaluating the effect on the shade of a dyestuff when dyed in the presence of metals used in the construction of dyeing machinery.

2. PRINCIPLE

2.1 The difference in shade between dyeings made in the presence and absence of salts of the metal is evaluated with the standard Grey Scale.

3. APPARATUS AND REAGENTS

3.1 Three pieces of undyed light wool serge of a size suitable for laboratory dyeing.

3.2 Three dye baths and solutions normal for the dyestuff.

3.3 Copper sulphate solution containing 0·2% $CuSO_4,5H_2O$ on the weight of one piece of the wool (See Section 3.1).

3.4 Ferric ammonium sulphate solution containing 0·5% $Fe_2(SO_4)_3.(NH_4)_2SO_4.24H_2O$ on the weight of one piece of the wool (See Section 3.1).

3.5 Grey Scale for evaluating change in colour (See Section 7.1).

4. SPECIMEN

4.1 (See Section 3.1).

5. PROCEDURE

5.1 Make three dyeings at the standard depth of shade on the wool serge according to the normal method for the dyestuff under examination. Before entering the fabric, add the ferric ammonium sulphate to one dye bath and the copper sulphate to another and bring the liquor ratio in each bath to 40 : 1.

5.2 Compare the shades of the dyeings made in the presence of copper sulphate and ferric sulphate with that of the dyeing made without and evaluate the differences with the Grey Scale.

6. REPORT

6.1 Report the dyestuff used, and method and strength of dyeing, and the numerical rating of the change in shade.

7. NOTES

7.1 See the following documents—

(*a*) Colour Fastness of Textiles: General Principles of Testing, ISO/TC 38/SC 1/31.

(*b*) Colour Fastness: Grey Scale for Evaluating Change in Colour, ISO/TC 38/SC 1/32.

ISO/TC 38/SC 1/45
Colour Fastness to Organic Solvents

Note— This draft, based on ISO/TC 38/SC 1/3, pages 9 and 10, has been prepared as a result of the discussions of the Subcommittee in New York on 10–12th November 1952. Following the meetings the draft was circulated in preliminary form to the delegates for comment and criticism and this final form includes the suggestions which have been received. It is now before the Subcommittee for discussion at its next meeting.

1. PURPOSE AND SCOPE

1.1 This method is intended for evaluating the resistance of the colour of textiles of all kinds in all forms to organic solvents, and in particular to solvents used in cleaning or mill processing.

2. PRINCIPLE

2.1 A specimen of the textile in contact with specified undyed cloths is agitated in the solvent. The change in colour of the specimen and staining of the undyed cloths are evaluated with standard Grey Scales.

3. APPARATUS AND REAGENTS

3.1 Suitable container with means of agitation (See Section 7.1).

3.2 Specified solvent (See Section 7.2).

3.3 Two undyed cloths, each 10 cm. × 4 cm., one piece made of the same kind of fibre as that in the textile to be tested, the second piece made of the fibre indicated below, or as otherwise specified (See Section 7.3).

If First Piece is—	Second Piece to be—
Cotton	Wool
Wool	Cotton
Silk	Cotton
Linen	Wool
Viscose	Wool
Acetate	Viscose
Polyamide	Wool

3.4 Grey Scales for evaluating change in colour and staining of undyed cloths (See Section 7.4).

4. SPECIMEN

4.1 If the textile to be tested is cloth, place a 10 cm. × 4 cm. specimen of it between the two pieces of white cloth required in Section 3.3, and sew along all four sides to form a composite specimen.

4.2 If the textile to be tested is yarn, knit it into cloth and treat it as in Section 4.1, or form a layer of parallel lengths of it between the two undyed cloths and sew around the four sides to hold the yarns in place.

4.3 If the textile to be tested is bulk fibre, comb and compress enough of it to form a sheet 10 cm. × 4 cm., place it between the two undyed cloths and sew around the four sides to hold the fibres in place.

5. PROCEDURE

5.1 Agitate the composite specimen continuously for 30 minutes in the solvent at 25°C. ±2°C. (77°F. ±4°F.) with a liquor ratio of 40 : 1. If the agitation is by hand, the specimen must be pressed against the container every two minutes with the stirring rod without removing the specimen from the solvent.

5.2 Squeeze excess solvent from the specimen and dry it in air at 80°C. ±2°C. (176°F. ±4°F.) without unstitching. Take the precautions necessary for safety in drying flammable or explosive solvents.

5.3 Remove the stitching and evaluate the change in colour of the specimen and the staining of the undyed cloths with the Grey Scale (See Section 3.4).

6. REPORT

6.1 Report the solvent used, the numerical rating for colour change, and the numerical rating for staining of each kind of white fibre tested.

7. NOTES

7.1 A 500 ml. beaker or other suitable open container may be used for the test, agitating by hand with a glass rod flattened at one end. A closed vessel agitated by shaking or tumbling in a hand or motor driven machine may be used.

7.2 It is anticipated that the test will be made in European countries with trichlorethylene and in the United States with perchlorethylene and with Stoddart Solvent (specially specified naphtha for use of dry cleaners). Other solvents for which information is desired may be used. Trichlorethylene should be used for rating dyestuffs.

Halogenated hydrocarbons should be stored over anhydrous sodium carbonate to neutralise hydrochloric acid.

7.3 The undyed cloths should be plain weave, medium weight, and free from finishes, residual chemicals, and chemically damaged fibres. Cotton and linen should be bleached, other materials should be cleaned to their usual degree of whiteness without bleaching. It is suggested that the kinds of fibres listed in Section 3.3 be rated regularly. Others may be rated in addition.

7.4 See the following documents—

(*a*) Colour Fastness of Textiles: General Principles of Testing, ISO/TC 38/SC 1/31.

(*b*) Colour Fastness: Grey Scale for Evaluating Change in Colour, ISO/TC 38/SC 1/32.

(*c*) Colour Fastness: Grey Scale for Evaluating Staining, ISO/TC 38/SC 1/33.

ISO/TC 38/SC 1/46

Colour Fastness to Potting

NOTE—This draft, based on ISO/TC 38/SC 1, page 19, has been prepared as a result of discussions of Subcommittee in New York, November 1951.

1. PURPOSE AND SCOPE

1.1 This method is intended for evaluating the resistance of the colour of wool and part wool textiles to the action of boiling water.

2. PRINCIPLE

2.1 A specimen of the textile between undyed wool and cotton cloths is rolled around a glass rod and treated with boiling water. The cloths are separated and dried, and the change in colour of the specimen and staining of the undyed cloths are assessed with standard Grey Scales.

3. APPARATUS AND REAGENTS

3.1 Vessel equipped with reflux to hold cylindrical specimen 4 cm. long in boiling water.

3.2 Glass rod 5 to 8 mm. in diameter.

3.3 Undyed, unbleached wool serge 10 cm. × 4 cm.

3.4 Undyed, bleached cotton limbric or the fibre used in mixture with the wool.

3.5 Grey Scales for evaluating change in colour and staining of undyed cloths (See Section 7.1).

4. SPECIMEN

4.1 If the textile to be tested is cloth, place a 10 cm. × 4 cm. specimen of it between the two pieces of undyed cloth required in Section 3.3 and 3.4 and sew along one of the shorter sides.

4.2 If the textile to be tested is yarn, knit it into cloth and treat it as in Section 4.1, or form a layer of parallel lengths of it between the two undyed cloths and sew around the four sides to hold the yarns in place.

4.3 If the textile to be tested is bulk fibre, comb and compress enough of it to form a sheet 10 cm. × 4 cm., place it between the two undyed cloths, and sew around the four sides to hold the fibres in place.

5. PROCEDURE

5.1 Roll the composite specimen around the glass rod to form a cylinder 4 cm. long and tie it loosely with thread.

5.2 Treat the specimen on the rod for 1 hour in boiling distilled water, liquor ratio 30 : 1, under reflux.

5.3 Remove the specimen from the rod and separate the undyed cloths from it. Dry all three in air at a temperature not exceeding 60°C. (140°F.).

5.4 Evaluate the change in colour of the specimen and the staining of the undyed cloths with the Grey Scales (See Section 7.1).

6. REPORT

6.1 Report the numerical rating for change in colour and the numerical rating for staining of each kind of undyed fibre tested.

7. NOTES

7.1 See the following documents—

(*a*) Colour Fastness of Textiles: General Principles of Testing, ISO/TC 38/SC 1/31.

(*b*) Colour Fastness: Grey Scale for Evaluating Change in Colour: ISO/TC 38/SC 1/32.

(*c*) Colour Fastness: Grey Scale for Evaluating Staining, ISO/TC 38/SC 1/33.

ISO/TC 38/SC 1/47

Colour Fastness to Pressing: Hot Pressing

NOTE—This draft, based on ISO/TC 38/SC 1/3, pages 11 to 13, has been prepared as a result of the discussions of the Subcommittee in New York on 10–12th November 1952. Following the meetings the draft was circulated in preliminary form to the delegates for comment and criticism and this final form includes the suggestions which have been received. It is now before the Subcommittee for discussion at its next meeting.

1. PURPOSE AND SCOPE

1.1 This method is intended for evaluating the resistance of the colour of textile yarns, thread, and fabrics of all kinds to ironing and to processing on hot cylinders.

1.2 Tests are given for hot pressing when the Textile is wet and when it is dry.

2. PRINCIPLE

2.1 For dry pressing, the dry specimen on a dry undyed cotton cloth is pressed with an iron of specified weight and temperature. For wet pressing, the wet specimen placed on a dry white cotton cloth and covered with a wet undyed cotton cloth is pressed with the iron. The change in colour and staining of the undyed cloths are evaluated with standard Grey Scales immediately and after a period of exposure to air.

3. APPARATUS AND REAGENTS

3.1 Hand iron of the proper weight to give a pressure of approximately 30 g./cm.² (See Section 7.1) at the temperature indicated in the following table (See Section 7.2).

Fabric	Temperature
Cotton and linen	190°C. to 210°C. (374°F. to 410°F.)
Wool, silk and rayon	140°C. to 160°C. (284°F. to 320°F.)
Acetate and polyamide	115°C. to 120°C. (239°F. to 248°F.)

3.2 Undyed, bleached, cotton cloth weighing approximately 125 g./m.² (3·7 oz./yd.²). Three pieces not less than 14 cm. × 4 cm. are required.

3.3 Grey Scales for evaluating change in colour and staining (See Section 7.3).

4. Specimen

4.1 If the textile to be tested is cloth, a piece 10 cm × 4 cm. is required.

4.2 If the textile to be tested is yarn or thread, knit it into cloth and use a piece 10 cm. × 4 cm. or wind it around a piece of thin cardboard 14 cm. × 4 cm. to obtain the area of the textile for test.

5. Procedure

5.1 Specimens of materials that have been subjected to any heat or drying treatment must be allowed to condition (by exposure to air at 65% relative humidity and 20°C. ± 2°C.) before they are tested.

5.2 Dry Pressing.— Place the specimen on the piece of dry cotton cloth (See Section 3.2) on a smooth horizontal surface. Place the iron on top and leave it for 10 seconds.

5.3 Wet Pressing.— Soak the specimen and a piece of the cotton cloth (See Section 3.2) in distilled water and squeeze or extract them to contain their own weights of water. Place the wet specimen on a piece of the dry cotton cloth (See Section 3.2) and place the wet cotton cloth on the specimen. Press by moving the iron to and fro over the wet cloth (without additional pressure) for 10 seconds.

5.4 Evaluate the change in colour of the specimen with the appropriate Grey Scale (See Section 7.3) immediately and again after the specimen has been allowed to condition for 4 hours in the standard atmosphere, i.e. 65% relative humidity and 20°C. ± 2°C. (68°F. ± 4°F.).

5.5 Evaluate the staining of the undyed cloths with the appropriate Grey Scale (See Section 7.3).

6. Report

6.1 Report the numerical rating for the change in colour for dry pressing and wet pressing immediately after testing and after conditioning for 4 hours. Report the numerical rating of the staining of the undyed cloth.

7. Notes

7.1 The pressure on the cloth under test will depend upon the area of the bottom of the iron lying over the 14 cm. × 4 cm. specimen.

7.2 [Method of measuring temperature of the iron to be supplied.]

7.3 See the following documents—

(*a*) Colour Fastness of Textiles: General Principles of Testing. ISO/TC 38/SC 1/31.

(*b*) Colour Fastness: Grey Scale for Evaluating Change in Colour. ISO/TC 38/SC 1/32.

(*c*) Colour Fastness: Grey Scale for Evaluating Staining. ISO/TC 38/SC 1/33.

ISO/TC 38/SC 1/48

Colour Fastness to Rubbing

Note— This draft, based on ISO/TC 38/SC 1/3, page 11, has been prepared as a result of discussions of the Sub-committee in New York, November 1952.

1. Purpose and Scope

1.1 This method is intended for evaluating the resistance of the colour of textile yarns, thread, and fabrics of all kinds to rubbing off and staining other materials.

1.2 Two tests are made, one with a dry rubbing cloth and one with a wet rubbing cloth.

2. Principle

2.1 Specimens of the textile are rubbed with dry undyed cotton cloth and with wet undyed cotton cloth. The staining of the undyed cloths is evaluated with the standard Grey Scale.

3. Apparatus and Reagents

3.1 Crockmeter or its equivalent (See Section 7.1).

3.2 Bleached undyed cotton lawn or similar cloth free from starch or other finish, cut into 5 cm. (2 inch) squares (See Section 7.2).

3.3 Grey Scale for evaluating staining of undyed cloth (See Section 7.3).

4. Specimen

4.1 If the textile to be tested is cloth, two pieces not less than 14 cm. × 5 cm. (5½ inch × 2 inch) are required for dry rubbing and two for wet rubbing. One specimen of each pair has the long direction parallel to the warp yarns, the other parallel to the filling yarns.

4.2 If the textile to be tested is yarn or thread knit it into cloth of suitable dimensions (See Section 4.1) or form a layer of parallel strands by wrapping it lengthwise on a cardboard rectangle of suitable dimensions.

5. Procedure

5.1 Dry Rubbing.— With the dry undyed cloth in place over the end of the finger of the testing device, rub it to-and-fro in a straight line over a track 10 cm. (4 inches) long on the dry specimen, 10 times in 10 seconds, with a downward force on the finger of 900 g. (2 pounds). Test warp and filling directions separately.

5.2 Wet Rubbing.— Repeat the test using fresh dry specimen and undyed cloth which have been wetted with water and squeezed to an add-on of 100%. Dry the cloths at room temperature.

5.3 Evaluate the staining of the undyed cloths with the Grey Scale (See Section 7.3).

6. Report

6.1 Report the numerical rating for dry staining and for wet staining for the direction, warp or filling, showing heavier staining.

7. Notes

7.1 The crockmeter may be obtained from the Calico Printers Association Ltd., St. James's Buildings, Oxford Street, Manchester, 1. It is described in the 1944 Year Book, A.A.T.C.C., page 192. Duplex crockmeters for testing two specimens simultaneously are described in *American Dyestuff Reporter*, Vol. 36, page 392 (1947).

7.2 Cloth for the purpose may be obtained from the Secretary, A.A.T.C.C., Lowell Technological Institute, Lowell, Massachusetts.

7.3 See the following documents—

(*a*) Colour Fastness: Grey Scale for Evaluating Staining, ISO/TC 38/SC 1/33.

(*b*) Colour Fastness of Textiles: General Principles of Testing, ISO/TC 38/SC 1/31.

ISO/TC 38/SC 1/49

Colour Fastness to Soda Boiling

NOTE—This draft, based on ISO/TC 38/SC 1/3, page 22, and ISO/TC 38/SC 1/15, page 2, has been prepared as a result of the discussions of the Subcommittee in New York on 10–12th November 1952. Following the meetings the draft was circulated in preliminary form to the delegates for comment and criticism and this final form includes the suggestions which have been received. It is now before the Subcommittee for discussion at its next meeting.

1. PURPOSE AND SCOPE

1.1 This method is intended for evaluating the resistance of the colour of natural and regenerated cellulose textiles in all forms to the action of boiling dilute sodium carbonate solutions.

1.2 Two tests are given, one with and the other without the addition of a reduction inhibitor.

2. PRINCIPLE

2.1 A specimen of the textile between specified undyed cloths is rolled around a glass rod and treated with boiling sodium carbonate solution with and without a reduction inhibitor. The composite specimen is rinsed, the cloths separated, dried, and the change in colour of specimen and staining of the undyed cloths are evaluated with standard Grey Scales.

3. APPARATUS AND REAGENTS

3.1 Vessel equipped with water-cooled reflux condenser to hold cylindrical specimen 4 cm. long in boiling solution.

3.2 Glass rod 5 to 8 mm. in diameter.

3.3 Desized undyed cotton fabric.

3.4 Undyed cloth of the type under test (or, if fibre or yarn is being tested, undyed cloth made from the same kind of fibre).

3.5 Sodium carbonate solution containing 10 g. anhydrous sodium carbonate per litre.

3.6 Sodium carbonate solution containing 10 g. anhydrous sodium carbonate per litre and 4 g. sodium *m*-nitrobenzenesulphonate per litre.

3.7 Grey Scales for evaluating changes in colour and staining of undyed cloths (See Section 7.1).

4. SPECIMEN

4.1 Two specimens prepared as follows, are required.

4.2 If the textile to be tested is cloth, place a 10 cm. × 4 cm. specimen of it between pieces of the undyed cloths specified in Section 3.3 and 3.4 and sew along one of the shorter sides.

4.3 If the textile to be tested is yarn, knit it into cloth and treat it as in Section 4.2, or form a layer of parallel lengths of it between the two undyed cloths and sew around the four sides to hold the yarns in place.

4.4 If the textile to be tested is bulk fibre, comb and compress enough of it to form a sheet 10 cm. × 4 cm., place it between the two undyed cloths, and sew around the four sides to hold the fibres in place.

5. PROCEDURE

5.1 Roll the composite specimen around the glass rod to form a cylinder 4 cm. long and tie it loosely with thread.

5.2 Treat one composite specimen on the rod for 1 hour in the boiling sodium carbonate solution, Section 3.5, liquor ratio 30 : 1, under reflux. Treat the other composite specimen in the same way and for the same time in boiling sodium carbonate solution containing the sodium *m*-nitrobenzenesulphonate, Section 3.6.

5.3 Remove the specimens from the rod, rinse for 10 minutes in cold running water, and separate the undyed cloths from them. Dry all pieces in air at a temperature not exceeding 60°C. (140°F.).

5.4 Evaluate the changes in colour of the specimens and the staining of the undyed cloths with the Grey Scales (See Section 7.1).

6. REPORT

6.1 Report the numerical ratings for change in colour and the numerical ratings for staining of each kind of undyed cloth tested with sodium carbonate alone and with sodium carbonate and sodium *m*-nitrobenzenesulphonate reduction inhibitor.

7. NOTES

7.1 See the following documents—

(*a*) Colour Fastness of Textiles: General Principles of Testing, ISO/TC 38/SC 1/31.

(*b*) Colour Fastness: Grey Scale for Evaluating Change in Colour, ISO/TC 38/SC 1/32.

(*c*) Colour Fastness: Grey Scale for Evaluating Staining, ISO/TC 38/SC 1/33.

ISO/TC 38/SC 1/50

Colour Fastness to Stoving

NOTE—This draft, based on ISO/TC 38/SC 1/3, page 21, has been prepared as a result of discussions of the Subcommittee in New York, November 1952.

1. PURPOSE AND SCOPE

1.1 This method is intended for evaluating the resistance of the colour of wool, silk, and mixed-fibre textiles containing one of these fibres, to the action of sulphur dioxide as used for bleaching animal fibres.

2. PRINCIPLE

2.1 A specimen of the textile containing its own weight of soap solution and a control are exposed in an atmosphere containing sulphur dioxide. The change in colour of the specimen is evaluated with a standard Grey Scale.

3. APPARATUS AND REAGENTS

3.1 Vessel of approximately 10 litres capacity for the sulphur dioxide atmosphere.

3.2 Sulphur.

3.3 Soap, containing not more than 5% moisture and complying with the following requirements based upon dry weight—

Free alkali calculated as Na_2CO_3	0·3% maximum
Free alkali calculated as NaOH	0·2% maximum (U.S.A.); 0·0% (S.D.C.)
Free fatty acid	0·1% maximum (U.S.A.); not to exceed 1% (S.D.C.)
Combined fatty acids calculated as Na salt	85% minimum
Titre of mixed fatty acids prepared from the soap ...	39 c. minimum
Iodine number of mixed fatty acids	To be supplied if desired
Acid number of mixed fatty acids	To be supplied if desired
pH of a 0·5% solution ...	9·5 to 10·5

3.4 Soap solution, containing 5 g. of soap (See Section 3.3) per litre of distilled water.

3.5 Control [To be supplied].

3.6 Grey Scale for evaluating change in colour (See Section 7.1).

4. SPECIMEN

4.1 If the textile to be tested is cloth, a piece at least 10 cm. × 4 cm. is required.

4.2 If the textile to be tested is yarn, knit it into cloth and use a piece at least 10 cm. × 4 cm. or from a number of lengths of it form a wick 10 cm. long and 0·5 cm. in diameter tied at both ends.

4.3 If the textile to be tested is bulk fibre, comb and compress enough of it to form a sheet at least 10 cm. × 4 cm.

5. PROCEDURE

5.1 Thoroughly impregnate the specimen and the test-control specimen by immersion for 5 minutes at 25°C. ± 2°C. (77°F. ± 4°F.) in the soap solution. Then squeeze so that each contains its own weight of solution.

5.2 Suspend the specimen and the test-control (See Section 3.5) for 16 hours in the vessel containing an atmosphere of sulphur dioxide obtained by igniting 5 g. of sulphur below the specimen and control and immediately closing the vessel.

5.3 Remove the specimen and control from the sulphur dioxide atmosphere and allow to hang in air for at least 2 hours without rinsing. The specimen and control are to be examined when dry. If the specimen contains cellulosic fibres, rinse it in cold running water and dry in air at a temperature not exceeding 60°C. (140°F.).

5.4 [Instructions for the control to be provided.]

5.5 Evaluate the change in colour of the specimen with the Grey Scale (See Section 7.1).

6. REPORT

6.1 Report the numerical rating for the colour change in the test.

7. NOTES

7.1 See the following documents—

(*a*) Colour Fastness of Textiles: General Principles of Testing. ISO/TC 38/SC 1/31.

(*b*) Colour Fastness: Grey Scale for Evaluating Change in Colour, ISO/TC 38/SC 1/32.

ISO/TC 38/SC 1/51

Colour Fastness to Washing: Handwashing

Note—This draft, based on ISO/TC 38/SC 1/3, has been prepared as a result of discussions of Subcommittee in New York, November 1952.

1. PURPOSE AND SCOPE

1.1 This method is intended for evaluating the resistance of the colour of textiles of all kinds in all forms to the action of soap solutions as used in washing them by hand.

2. PRINCIPLE

2.1 A specimen of the textile in contact with specified undyed cloths is agitated in a soap solution, rinsed, and dried. The change in colour of the specimen and staining of the undyed cloths are evaluated with standard Grey Scales.

3. APPARATUS AND REAGENTS

3.1 Suitable container with means of agitation (See Section 7.1).

3.2 Soap, containing not more than 5% moisture and complying with the following requirements based upon dry weight—

Free alkali calculated as Na_2CO_3	0·3% maximum
Free alkali calculated as NaOH	0·2% maximum (U.S.A.); 0·0% (S.D.C.)
Free fatty acid	0·1% maximum (U.S.A.); not to exceed 1% (S.D.C.)
Combined fatty acids calculated as Na salt	85% minimum
Titre of mixed fatty acids prepared from the soap	39 c. minimum
Iodine number of mixed fatty acids	To be supplied if desired
Acid number of mixed fatty acids	To be supplied if desired
pH of a 0·5% solution ...	9·5 to 10·5

3.3 Soap solution, containing 5 g. of soap (See Section 3.2) per litre of distilled water.

3.4 Two undyed cloths, each 10 cm × 4 cm., one piece made of the same kind of fibre as that in the textile to be tested, the second piece made of the fibre indicated below, or as otherwise specified.

If First Piece is—	Second Piece to be—
Cotton	Wool
Wool	Cotton
Silk	Cotton
Linen	Wool
Viscose	Wool
Acetate	Viscose
Polyamide	Wool

3.5 Grey Scales for evaluating change in colour and staining of undyed cloths (See Section 7.2).

4. SPECIMEN

4.1 If the textile to be tested is cloth, place a 10 cm. × 4 cm. specimen of it between the two pieces of undyed cloth required in Section 3.4, and sew along all four sides to form a composite specimen.

4.2 If the textile to be tested is yarn, knit it into cloth and treat it as in Section 4.1, or form a

layer of parallel lengths of it between the two undyed cloths and sew around the four sides to hold the yarns in place.

4.3 If the textile to be tested is bulk fibre, comb and compress enough of it to form a sheet 10 cm. × 4 cm., place it between the two undyed cloths, and sew around the four sides to hold the fibres in place.

5. PROCEDURE

5.1 Agitate the composite specimen continuously for 30 minutes in the soap solution at 40°C. ±2°C. (104°F. ±4°F.) with a liquor ratio of 50 : 1. If the agitation is by hand, press the specimen against the container 10 times every three minutes without removing it from the solution. If mechanical agitation is used (See Section 7.1) the time of treatment and if necessary liquor ratio shall be adjusted so as to give results identical with those of the hand test.

5.2 Rinse the composite specimen twice in distilled water and then for 10 minutes in cold running water. Squeeze it, remove the stitching along three sides, and dry in air at a temperature not over 60°C. (140°F.), with the three parts in contact only along the one row of stitching.

5.3 Evaluate the change in colour of the specimen and the staining of the undyed cloths with the Grey Scales (See Section 7.2).

6. REPORT

6.1 Report the numerical ratings for colour change and for staining of each kind of undyed fibre tested.

7. NOTES

7.1 A 500 ml. beaker or other suitable open container may be used for the test, agitating by hand with a glass rod flattened at one end. A closed vessel agitated by shaking or tumbling it by hand, or a motor driven machine may be used.

7.2 See the following documents—

(*a*) Colour Fastness of Textiles: General Principles of Testing, ISO/TC 38/SC 1/31.

(*b*) Colour Fastness: Grey Scale for Evaluating Change in Colour, ISO/TC 38/SC 1/32.

(*c*) Colour Fastness: Grey Scale for Evaluating Staining, ISO/TC 38/SC 1/33.

ISO/TC 38/SC 1/52

Colour Fastness to Water, Flat Dish Method

NOTE—This draft, based on ISO/TC 38/SC 1/3, pages 2 and 3, has been prepared as a result of the discussions of the Subcommittee in New York on 10–12th November 1952. Following the meetings the draft was circulated in preliminary form to the delegates for comment and criticism and this final form includes the suggestions which have been received. It is now before the Subcommittee for discussion at its next meeting.

1. PURPOSE AND SCOPE

1.1 This method is intended for evaluating the resistance of the colour of textiles of all kinds in all forms to immersion in water.

2. PRINCIPLE

2.1 A specimen of the textile in contact with specified undyed cloths is covered with water in a flat dish and then with a glass plate. The water is poured off and the wet cloths allowed to lie under the glass. The specimen and undyed cloths are dried separately. The change in colour of the specimen and staining of the undyed cloths are evaluated with standard Grey Scales.

3. APPARATUS AND REAGENTS

3.1 Flat-bottomed dish, area of bottom greater that 5 cm. × 5 cm.

3.2 Smooth glass plate 5 cm. × 5 cm., or slightly larger weighing approximately 50 g.

3.3 Two undyed cloths, each 5 cm × 5 cm. One piece made of the same kind of fibre as that in the textile to be tested, the second piece made of the fibre indicated below, or as otherwise specified (See Section 7.1).

If First Piece is—	Second Piece to be—
Cotton	Wool
Wool	Cotton
Silk	Cotton
Linen	Wool
Viscose	Wool
Acetate	Viscose
Polyamide	Wool

3.4 Standard Grey Scales (See Section 7.2).

3.5 Oven maintained at 37°C. ± 2°C. (100°F. ±4°F.).

4. SPECIMEN

4.1 If the textile to be tested is cloth, place a 5 cm. × 5 cm. specimen of it between the two pieces of the white cloth, Section 3.3, and sew along one side to form a composite specimen.

4.2 If the textile to be tested is yarn, knit it into cloth and treat it as in Section 4.1 or form a layer of parallel lengths of it between the two undyed cloths and sew along two opposite sides near the ends of the yarns to form a composite specimen.

4.3 If the textile to be tested is fibre, comb and compress some of it into a sheet 5 cm. × 5 cm. in size, place the sheet between the two undyed cloths, and sew along two opposite sides to form a composite specimen.

5. PROCEDURE

5.1 Thoroughly wet the composite specimen in distilled water at 25°C. ±2°C. (77°F. ±4°F.). Lay it out smooth in the flat-bottomed dish and cover it with distilled water. Place the glass plate on it and press evenly and lightly with three fingers to remove air bubbles. Allow to stand for 15 minutes at 25°C. ±2°C. (77°F. ±4°F.).

5.2 Pour off the water without removing the glass plate. Allow the specimen to remain under the plate for 4 hours at 37°C. ±2°C. (98°F. ±4°F.).

5.3 Separate the specimen and white cloths and dry them apart in air at a temperature not exceeding 60°C. (140°F.).

5.4 Evaluate the change in colour of the specimen and the staining of the undyed cloths with the Grey Scales (See Section 7.2).

6. REPORT

6.1 Report the numerical rating for colour change and the numerical rating for staining of each kind of undyed fibre tested.

7. NOTES

7.1 See the first reference in Section 7.2 below, especially Section 7.8 in it.

7.2 See the following documents—

(*a*) Colour Fastness of Textiles: General Principles of Testing, ISO/TC 38/SC 1/31.

(*b*) Colour Fastness: Grey Scale for Evaluating Change in Colour, ISO/TC 38/SC 1/32.

(*c*) Colour Fastness: Grey Scale for Evaluating Staining, ISO/TC 38/SC 1/33.

ISO/TC 38/SC 1/53
Colour Fastness to Water, Test Tube Method

NOTE—This draft, based on ISO/TC 38/SC 1/3, pages 2 and 3, has been prepared as a result of the discussions of the Subcommittee in New York on 10–12th November 1952. Following the meetings the draft was circulated in preliminary form to the delegates for comment and criticism and this final form includes the suggestions which have been received. It is now before the Subcommittee for discussion at its next meeting.

This method is similar to that for "Colour Fastness to Water, Flat Dish Method" ISO/TC 38/SC 1/52, except that the composite specimen is rolled into a cylinder and treated in a test tube of suitable dimensions to take the roll easily instead of in the flat dish, liquor ratio 6 : 1, and the water is not poured off.

ISO/TC 38/SC 1/54
Colour Fastness to Water, Perspirometer Method

This method is similar to that for "Colour Fastness to Water, Flat Dish Method" ISO/TC 38/SC 1/52, except that the specimens and undyed cloths are about 6·5 cm. (2½ inches) square, and after immersion in water at room temperature for 15 minutes and pouring off the water, the composite specimen is placed between two glass plates measuring about 7·5 × 6·5 cm. (3 × 2½ inches) under a force of about 4·5 kg. (10 lb.) for the 4 hours at 37°C. ±2°C. (100°F. ±4°F.).

NOTE—The "Perspirometer" developed by the American Association of Textile Chemists and Colorists is intended for carrying out tests of this kind. For information about it write to The Secretary, A.A.T.C.C., Lowell Technological Institute, Lowell, Massachusetts.

ISO/TC 38/SC 1/55
Colour Fastness to Sea Water, Flat Dish Method

NOTE—This draft, based on ISO/TC 38/SC 1/3, pages 5 and 6, has been prepared as a result of discussions of Subcommittee in New York, November 1952.

This method is similar to that for "Colour Fastness to Water, Flat Dish Method" ISO/TC 38/SC 1/52, except that distilled water containing 30 g. of sodium chloride per litre is used instead of distilled water.

ISO/TC 38/SC 1/56
Colour Fastness to Sea Water, Test Tube Method

This method is similar to that for "Colour Fastness to Water, Test Tube Method" ISO/TC 38/SC 1/53, except that distilled water containing 30 g. of sodium chloride per litre is used instead of distilled water, the liquor ratio is 6 : 1, and the liquor is not poured off.

ISO/TC 38/SC 1/57
Colour Fastness to Sea Water, Perspirometer Method

NOTE—This draft, based on ISO/TC 38/SC 1/3, pages 5 and 6, has been prepared as a result of the discussions of the Subcommittee in New York on 10–12th November 1952. Following the meetings the draft was circulated in preliminary form to the delegates for comment and criticism and this final form includes the suggestions which have been received. It is now before the Subcommittee for discussion at its next meeting.

This method is similar to that for "Colour Fastness to Water, Perspirometer Method" ISO/TC 38/SC 1/54, except that distilled water containing 30 g. of sodium chloride per litre is used instead of distilled water.

ISO/TC 38/SC 1/58
Colour Fastness to Water Spotting

NOTE—This draft, based on ISO/TC 38/SC 1/3, page 4, has been prepared as a result of discussions of subcommittee in New York, November 1952.

1. PURPOSE AND SCOPE

1.1 This method is intended for evaluating the resistance of the colour of textiles of all kinds in all forms to spotting by water.

2. PRINCIPLE

2.1 A droplet of distilled water is worked into the textile with a glass rod and the change in colour of the textile is evaluated with a standard Grey Scale after two minutes and after drying.

3. APPARATUS AND REAGENTS

3.1 Pipette or dropper to deliver approximately 0·15 ml. of water.

3.2 Glass rod with rounded end.

3.3 Grey Scale for evaluating change in colour (See Section 7.1).

4. SPECIMEN

4.1 If the textile to be tested is cloth, a piece at least 10 cm. × 4 cm. is required.

4.2 If the textile to be tested is yarn, knit it into cloth and use a piece at least 10 cm. × 4 cm. or form a wick from a number of lengths of it, about 10 cm. long and 0·5 cm. in diameter, tied at both ends.

4.3 If the textile to be tested is fibre, comb and compress some of it into a sheet at least 10 cm. × 4 cm. in size.

5. PROCEDURE

5.1 Spot the specimen with approximately 0·15 ml. of distilled water at 25°C. ±2°C. (78°F. ±4°F.). Work the droplet into the specimen with the glass rod.

5.2 After 2 minutes evaluate the change in colour in the periphery of the spot with the Grey Scale.

5.3 Dry the specimen in air at 25°C. ±2°C. (78°F. ±4°F.) and evaluate the change in colour in the periphery of the spot with the Grey Scale.

6. REPORT

6.1 Report the numerical rating for change in colour after 2 minutes and after drying.

7. NOTES

7.1 See the following documents—

(*a*) Colour Fastness of Textiles: General Principles of Testing, ISO/TC 38/SC 1/31.

(*b*) Colour Fastness: Grey Scale for Evaluating Change in Colour, ISO/TC 38/SC 1/32.

(*Received 1st October 1953*)

COMMUNICATIONS

Observations on some Commercial Colour-matching Lamps

J. C. GUTHRIE and P. H. OLIVER

Four commercial colour-matching lamps of the fluorescent-tube type have been considered. Differences exist between the lamps and Illuminant C, and also lesser differences between the lamps themselves. Samples vary in hue from lamp to lamp, but no serious discrepancies have been found in colour matching: i.e. a match under one lamp is usually a match under one of the others, since in a large number of cases the dyeing has been found to have a similar spectral curve to the pattern. Daylight is very variable, and it is doubtful whether a single standard colour-matching lamp would be advisable.

The merits of a universally acceptable standard light source for colour matching are attractive at first sight, but the existing British Standard [1] does not specify a single source: it merely "covers sources of light so designed that the light emitted by them is sufficiently close to daylight in both colour and spectral energy distribution to enable these sources to be used for colour matching or similar purposes". The requirements include the condition that the colour of the light given by a colour-matching fitting shall fall within the limits indicated by the area *EFGH* on the C.I.E. colour chart in Fig. 1. In addition, it is necessary for the spectral energy distribution to satisfy certain other requirements.

Colour-matching lamps are usually mounted in the roof of an enclosed cabinet, and Schofield [2] has described a cabinet designed for use with painted surfaces. Hewitt [3] has suggested viewing conditions for optimum effect with lustrous yarns, dark shades, etc. A simple type of cabinet which has proved satisfactory in the Droylsden routine laboratory is approx. 10 ft. × 6 ft. and 8 ft. high with the roof and walls white. Fluorescent colour-matching tubes are fixed to the roof, and dyeings are examined on a black shelf about 3 ft. from the floor, near which the light intensity is about 120 ft.-candles. This cabinet is used for examining patterns and matchings.

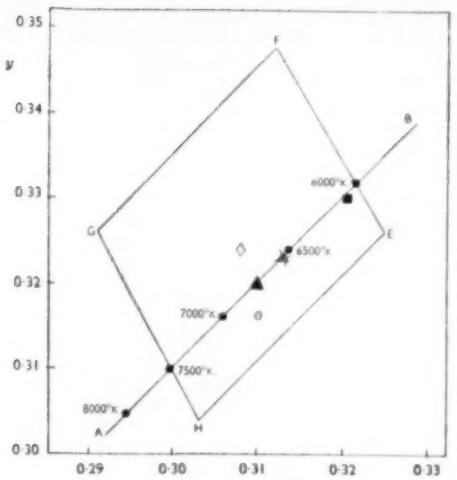
Colour-matching lamps from different manufacturers are not exactly alike, and if the lamps in the two halves of a cabinet are, say, Mazda and Osram respectively, the colours of the light in the two halves are visibly quite different. It is necessary to know how the variations between lamps affect the appearance of textiles, and also whether colour-matching judgments are affected.

Colour-matching tubes of the fluorescent type have been described by others [4,5]. Briefly, light is provided by an arc discharge through mercury vapour, the spectral characteristics depending on the phosphors chosen to coat the inside of the tube. The term *phosphor* covers a wide range of inorganic substances which emit in the visible part of the spectrum when subjected to ultraviolet radiation. Organic materials are not used because of the high-temperature (about 400°C.) treatment in the manufacture of the tubes.

The tubes are usually specified by the manufacturer in terms of (i) trichromatic coefficients on the C.I.E. system, (ii) luminance in each of eight wavebands, and (iii) colour temperature; (i) and (ii) are in accordance with recommendations made by the 1948 meeting of the C.I.E. [6]. Luminance is obtained from the spectral energy curve by weighting values along the curve with published data for the standard observer at each wavelength. The "standard observer" data [7,8] were specified at the 1931 meeting of C.I.E., and luminance therefore defines the lamps as the average eye sees them. The colour temperature of light sources and methods of measurement have been discussed by Harding [9]. Attempts were made to measure the spectral energy distributions of lamps supplied by three of the manufacturers—Mazda, Osram, and Atlas. These were obtained in two types—5 ft., 80 watt and 2 ft., 40 watt. The measurements were made on a Hilger Uvispek photoelectric spectrophotometer, the lamp being mounted above the instrument and the light from it led through the entrance slits. Comparison measurements were made with the tungsten lamp housed in the spectrophotometer. Results, however, were not found to be sufficiently reproducible and did not appear to be in very good agreement with the manufacturers' data. A possible cause of some of the errors is the presence of the mercury line spectrum, and discrepancies could be caused by the assumptions regarding the energy distribution of the tungsten lamp. Also the measurements were on single lamps and no measure of variation between lamps was possible.

In the following calculations the manufacturers' data have therefore been used for Mazda, Siemens, and Atlas lamps. The manufacturers' data for Mazda and Osram were identical, both makers being members of the Electric Lamp Manufacturers Association and all information in the tables and figures quoted for Mazda apply also to Osram. To the eye these lamps appeared to be quite different, the Osram appearing definitely redder. The data for the General Electric Company's 80-watt colour-matching lamp MCF/U have been taken from Winch and Young [10] and are actual measurements as distinct from the manufacturers' objectives. The Siemens lamp is a combination of fluorescent tubes and tungsten lamps and the relative merits of such a lamp have been discussed by Harrison [11].

Trichromatic coefficients for the lamps (excluding the G.E.C. MCF/U) have been plotted in Fig. 1 together with the National Physical Laboratory figures for mean north skylight and mean noon sunlight. Illuminant A [12–14] has a colour temperature of 2848°K. with C.I.E. coordinates (0·448, 0·407, 0·145). This approximates to the usual tungsten lamp, which is therefore well removed from the permissible area *EFGH* for colour-matching fittings.



x	y	
0·310	0·320	▲ Siemens
0·313	0·323	⁄ Atlas
0·313	0·323	△ Mazda
0·308	0·324	⊹ Mean north skylight (N.P.L.* average)
0·3204	0·3301	■ Mean noon sunlight (N.P.L.* average)
0·3101	0·3163	◉ Illuminant C

AB Locus of colour temperature

FIG. 1—C.I.E. Colour Chart

* National Physical Laboratory

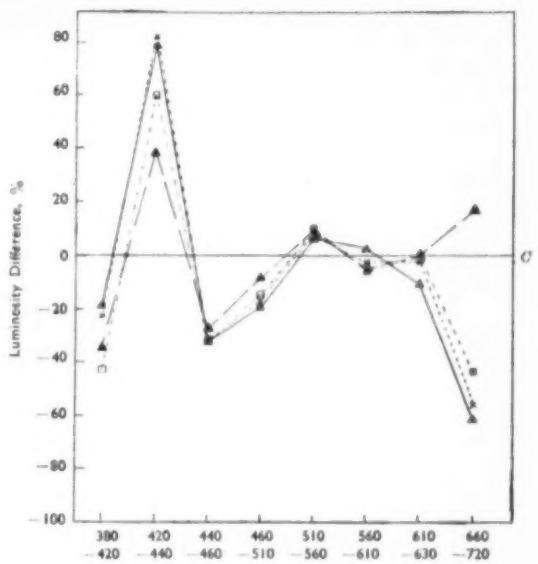


FIG. 2

TABLE I

Percentage Luminance

Waveband, mμ.	Mazda	G.E.C. MCF/U	Siemens	Atlas	Illuminant C
380–420 Far violet ...	0·021	0·015	0·017	0·02	0·026
420–440 Violet ...	0·47	0·42	0·365	0·48	0·263
440–460 Blue	0·62	0·62	0·66	0·62	0·909
460–510 Blue–green ...	9·0	9·5	10·2	9·2	11·103
510–560 Green ...	43·6	44·9	44·5	44·35	40·86
560–610 Yellow ...	37·1	34·3	34·4	35·3	36·15
610–660 Light red ...	8·9	9·9	10·0	9·7	9·95
660–760 Far red ...	0·28	0·41	0·85	0·32	0·726

The luminance figures (Table I) were compared with published data on Illuminant C [13,14], which is an approximation to average daylight, and Fig. 2 shows the difference between each lamp and Illuminant C for each of the wavebands. It was not possible to confirm that these lamps comply with *B.S.* 950 [1], Section 7, since the necessary filters were not available. The lamps would possibly fail owing to deficiency in red compared with Illuminant C, i.e. with filters 8 and 9 in the *B.S.* 950 test; and the excess in violet, due to a strong mercury line, is not effectively countered by fairly large deficiencies in the bands on either side, i.e. far violet and blue.

The differences between the lamps themselves are generally less than the differences between any one lamp and Illuminant C. Compared with Illuminant C, all lamps are deficient in far violet, all show a surplus in violet, all are deficient in blue and blue–green. The green and yellow bands show small differences, some lamps are equal to Illuminant C in the red, and except for the Siemens type all are deficient in far red. The surplus in violet is due to the presence of a powerful mercury line at 436 mμ., and the deficiencies in the far violet and the blue appear to be an attempt to compensate for this. The Siemens type is rich in far red, owing to the presence of tungsten lamps as well as fluorescent tubes. Whilst Illuminant C is an internationally agreed standard, it is not necessarily correct to assume that it is a good representative of daylight. This point will be referred to again in a later section.

VARIATION IN CHROMATICITY COORDINATES OF A DYED TEXTILE WITH TYPE OF ILLUMINANT

Reflectance curves of red, apple green, and light blue from the spun-coloured Fibro range [15] were plotted, and trichromatic coefficients were calculated for each colour, each lamp being used in turn as the reference light source. The results have been plotted on the uniform-chromaticity diagrams shown in Fig. 3–5. The "weighted ordinate" method was used to evaluate the tristimulus values—

$$X = \int RE\bar{x}d\lambda\,, \qquad Y = \int RE\bar{y}d\lambda\,, \qquad Z = \int RE\bar{z}d\lambda$$

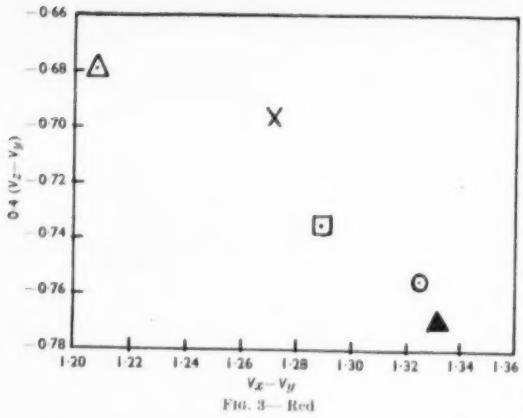


FIG. 3—Red

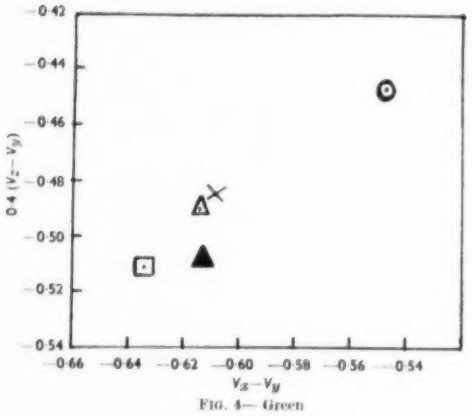


FIG. 4—Green

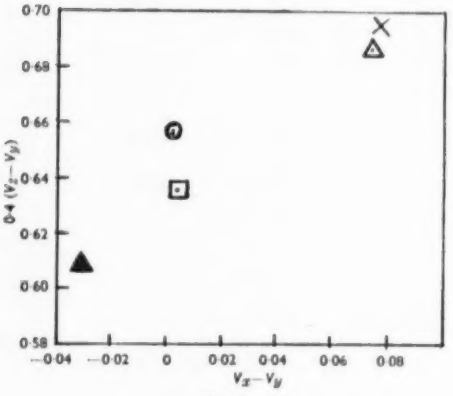


FIG. 5—Blue

(R = reflectance; E = energy of the illuminant; and $\bar{x}$, $\bar{y}$, $\bar{z}$ are distribution coefficients) [16]. These integrals reduce to summations of the mean values for each waveband, taken over the visible spectrum. The data in Table I are effectively values of Ey, so that Y is obtained at once, since R is known.

As might be expected from Fig. 1, the plots for the Mazda and Atlas lamps are close together, although this is not so obvious for the red Fibro. These two lamps give figures nearer Illuminant C than do the G.E.C. MCF/U and Siemens for apple green, but the reverse is true for the red and light blue shades.

The chromaticity diagram tends to be a little misleading as regards the visual impressions of colour differences, since the locus of minimum perceptible colour differences plotted about any point in the diagram is not a circle, as might be expected, but in general is an ellipse, with the smallest ellipses in the blue region and the largest in the green [17]. To assess the actual visual impression of colour differences on the coloured samples under the various illuminants, the C.I.E. coordinates have been transformed to Adams "chromatic value" space, and the number of units of colour difference evaluated by the following formula, quoted by Nickerson [18]—

$$\Delta E = 40 \left\{ (0{\cdot}23\,\Delta V_y)^2 + \left[\Delta(V_x - V_y)\right]^2 + \left[0{\cdot}4\,\Delta(V_z - V_y)\right]^2 \right\}^{\frac{1}{2}}$$

where V_x, V_y, and V_z are Munsell-value functions of the chromaticity coordinates which have been tabulated in terms of X, Y, and Z respectively.

When used as a measure of fading of the Society's light fastness standards [19], this formula has been queried by Ricketts [20], but Nickerson [18] has produced evidence of its reliability for small values of ΔE. Its validity is therefore not rigidly established, but it is probably the best quantitative expression which has been put forward for such small colour differences as are being considered. The factor 40 is included to make one unit approximately equal to a just perceptible colour step.

Results obtained on the colour of the samples under the different lamps compared with their colour under Illuminant C (Table II) confirm the qualitative statements previously made.

TABLE II

Differences between Each Lamp and Illuminant C for Three Spun-coloured Samples of Fibro

Colour	ΔE calculated from the Adams formula			
	Mazda	G.E.C. MCF/U	Siemens	Atlas
Red	7·8	3·85	1·8	5·2
Apple green ...	2·2	3·32	2·6	1·9
Light blue ...	3·7	0·88	1·8	4·1

A similar series of measurements has been made by Winch and Young [10], who discussed the objective chromaticity shifts of a series of objects illuminated by several fluorescent lamps. They dealt with several main classes of lighting and included one colour-matching tube. These chromaticity shifts were directly related to the energy distributions of the light sources. Wright [21] has warned of the danger of judging the colour-rendering properties of an illuminant solely from chromaticity data. The chromaticity shift may be large and yet appear small to the eye, since the adaptation of the eye tends to compensate for any change in chromaticity of the illumination. This has been referred to also by Godlove [22]. In order to eliminate this effect when viewing by eye, a special twin illuminating box was used, so that

samples could be viewed simultaneously under two different lamps.

COLOUR MATCHING

Since the colour of a yarn or a fabric may change when viewed under these different lamps, it is important to know whether a match under one illuminant will still be a match when looked at under another. If the dyeing reproduces the spectral reflectance curve of the pattern, then it will not matter what illuminant is used, but if the match has been obtained by equalising the tri-chromatic coefficients (a necessary condition for a match), then the reflectance curves will not necessarily coincide and the choice of illuminants will be important. White and Vickerstaff have explained this point in the second Mercer lecture [23].

A large number of dyeings have been examined, and reflectance curves plotted for both pattern and dyeing in each case. The dyeings were matched in the routine laboratory in the normal way, and it was noticeable that the curves for the dyeing and the pattern were nearly always close to each other. This is probably due to the fact that the match was looked at in several different lights, such as early morning northlight, noon northlight, and under colour-matching lamps, and the dyer was, either consciously or otherwise, ensuring that the spectral reflectance curves of dyeing and pattern were close together. In order to obtain quantitative data on the difference between the dyeing and the pattern the Adams formula [18] for small colour differences was applied to reflectance curves obtained on the Uvispek.

TABLE III

Chromaticity Differences and Colour Matching

(All dyeings were on Courtaulds continuous-filament viscose rayon yarn 150/27)

(*a*) Pattern (*b*) Dyeing	ΔE between Pattern and Dyeing				
	Illuminant C	Mazda	G.E.C. Siemens MCF/U	Atlas	
BLUE					
(*a*) Unknown...	1·2	1·4	1·2	1·2	1·2
(*b*) Match ...					
DARK BLUE					
(*a*) Unknown...	5·8	5·5	5·8	5·3	5·5
(*b*) Near match					
FAWN					
(*a*) Direct dyed	1·7	1·8	1·7	1·7	1·8
(*b*) Vat dyed...					
YELLOW					
(*a*) Unknown...	6·5	6·5	6·3	6·4	6·6
(*b*) Near match					

Table III contains typical results and includes samples in which a match was obtained and also others where a good match had not occurred. The latter would correspond to an intermediate match on the way to obtaining the final acceptable match. Fig. 6 and 7 show the spectral reflectance curves of two of the above matchings, and Fig. 8 and 9 show the plots on the uniform chromaticity scale for each of the lamps.

Values of ΔE of the order of 1–1·5 would be a match. It is appreciated that high accuracy cannot be claimed, since eight broad wavebands have been considered, and the following conclusions are put

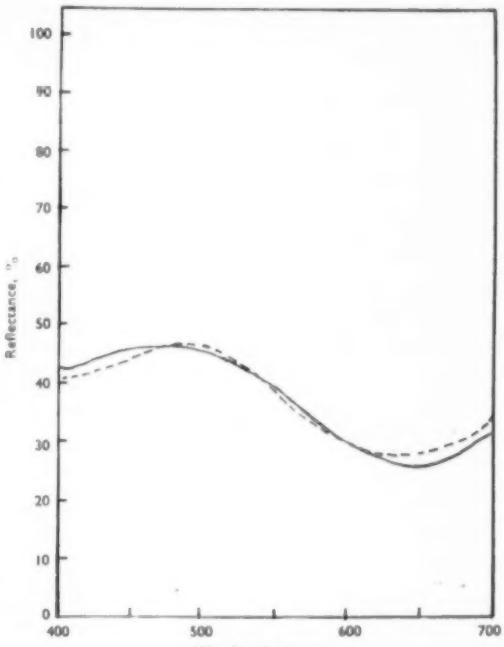


FIG. 6—Blue

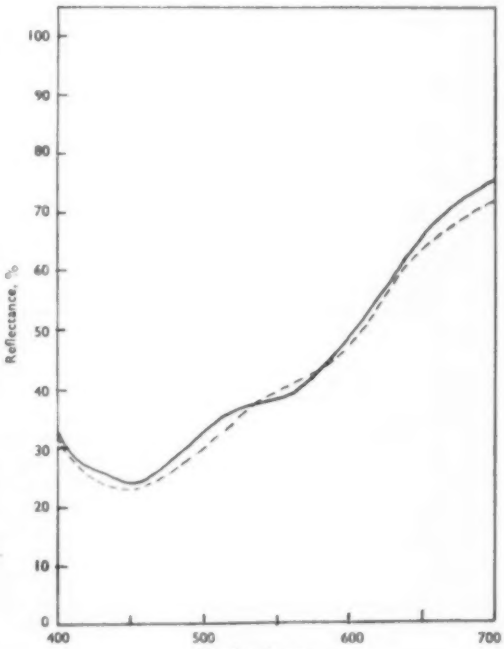


FIG. 7—Fawn

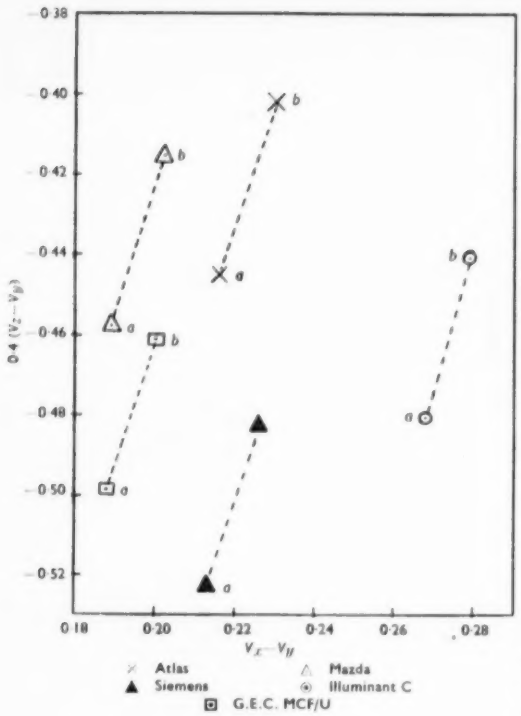


FIG. 8— Fawn

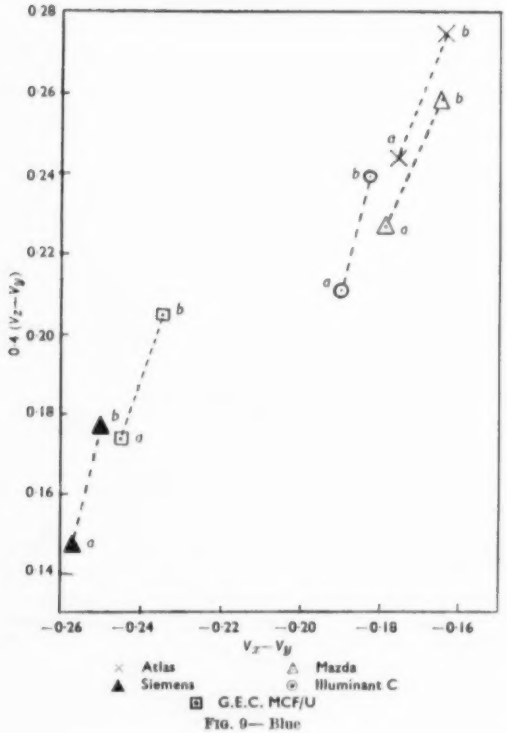


FIG. 9— Blue

forward tentatively. The illuminant appears to have less effect on the match than might be expected, and in almost every case the values of ΔE do not show significant variations from lamp to lamp. Also this is an indirect confirmation of the fact that many matches are made by fairly close reproduction of the spectral reflectance curve of the pattern. It has been shown how the colour of a sample changes from one illuminant to another and since the dyeing is showing a similar reflectance curve to the pattern in most cases, the dyeing and the pattern will change by similar amounts from one illuminant to another, and so match every time.

Daylight itself is very variable, as shown in Fig. 1, and it is obviously important to try and reproduce the reflectance curves when matching a pattern. Cunliffe [24] has shown how daylight can vary at different times of day and on different days. His data show the variation that can occur between an overcast sky, one with drifting clouds, and north sky at a given time of the year, north sky being relatively bluer than either. Also daylight varies on a given day, being redder in the early morning and late afternoon than at midday. Harrison [11] has compared the spectral curves of Illuminant C and overcast sky at a colour temperature of 6500°K., and has shown appreciable differences to exist between the curves. North daylight is considered by most colour matchers to be the best form of daylight illumination. This lighting is chosen presumably to avoid direct sunlight illumination and on the assumption that it is less subject to spectral quality variations. Measurements substantiating this point seem, however, to be lacking.

Taylor and Kerr [25] have made a series of measurements of spectral distributions and colour temperature of sunlight, sky light, and combinations of both.

TABLE IV

Colour Temperatures of Daylight

Condition of Viewing	Colour Temperature (°K.)
North sky, no clouds but hazy	8,770–12,600
Zenith sky, no clouds	8,800–60,000
Sun plus sky, incident on horizontal surface	5,960– 6,100
Overcast sky	6,490– 6,510

Their figures (Table IV) indicate a much greater variation in the qualities of north sky lighting than for illumination by a combination of sun and sky light. It should also be noted that the figure 6500°K. (used in Great Britain) appears to be on the low side as an average colour temperature for north sky light if compared with the above figures. If north sky light is accepted for colour matching, a higher colour temperature is required for the U.S.A. than for this country, and the American figures would not be applicable to Great Britain [26].

For commercial purposes it might be thought advisable to specify a colour-matching source more exactly and a match under this would be the final match. As stated above, it would be possible for matches to be made according to this lamp which would not match under other illuminants (e.g. the various kinds of daylight). A customer whose dyeing did not match the pattern under the

daylight in which he viewed would probably not be satisfied by being told that a match would be obtained under the standard lamp.

It may be concluded that, although commercial fluorescent colour-matching lamps vary from maker to maker, the differences are not serious for commercial colour matchings. Hue differences can be detected when dyeings are viewed under different illuminants, but in many cases dyeing and pattern change in a similar way, showing that the spectral reflectance curves have been fairly closely reproduced.

* * *

Data on the lamps were supplied by the General Electric Co. Ltd. (Osram), the British Thomson-Houston Co. Ltd. (Mazda), Siemens Electric Lamps & Supplies Ltd., and Thorn Electrical Industries Ltd. (Atlas). Reflectance curves of the three spun-coloured Fibro samples were supplied by Messrs. Imperial Chemical Industries Ltd. (Blackley).

The experimental work and colour calculations were carried out by Mrs. B. M. Hyde.

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Some Experiments on the Overdyeing of Nylon

C. D. O'BRIAIN and R. H. PETERS

Rate-of-dyeing curves have been measured for nylon dyed under very acid conditions (pH ca. 1·5). The curve falls into two parts; in the initial stages the uptake is rapid, and this is followed by a slow increase in dye concentration which does not reach a maximum. By stripping the dye from the fibre and redyeing under conditions of high pH, the number of free amine ends has been estimated. From the results it has been shown that during the second part of the rate-of-dyeing curves hydrolysis occurs, giving an increased number of amine groups. The amount of dye taken up at the low pH values is, however, far in excess of that required to saturate the amine groups and must be accounted for by "amide group" dyeing. Viscosities have also been used to confirm the hydrolysis.

The picture of the dyeing process at low pH is, therefore, initial absorption of dye on to sites provided by the peptide groups either as the free dye acid or by absorption of the hydrogen ions in the dyebath followed by the dye anion. Simultaneous with the absorption is a slow but definite hydrolysis of the nylon molecules with consequent degradation of the fibre.

INTRODUCTION

Because of their good fastness properties, acid dyes have been employed to a considerable extent in the dyeing of nylon. Unfortunately, they exhibit, when dyed in mixtures under normal conditions of acid concentration (pH = 3–4, say), incompatibility to an extreme degree. It is for reasons such as these that studies of the dyeing mechanism are important and have proved of value in overcoming practical dyeing difficulties. The work has been discussed in some detail by Vickerstaff [1] and has led to the following picture of the dyeing process. At high pH, the dye is adsorbed on amino groups in the nylon in a manner analogous to the dyeing of wool. The titration curve which represents the amount of dye adsorbed as a function of pH has been given by Peters [2]. The initial adsorption at high pH yields the usual **S**-shaped curve, and soon reaches a constant value with decreasing pH, this value corresponding to saturation of the amino groups present in the nylon. When the pH falls to 2·5–3·5, further adsorption occurs, which increases rapidly with further decrease in pH and shows no sign of reaching a maximum. Although, at first sight, the titration curve of nylon is different from those of wool and silk, recent work by Elöd and Fröhlich [3] on the adsorption of hydrochloric, monochloroacetic, acetic, and formic acids has shown a formal similarity between the titration curves of the three fibres. Wool and silk have a considerably larger number of amino groups than nylon but, when these are saturated,

it is possible by further decrease in pH to cause the fibre to adsorb more acid. The differences between the titration curves of different dyes lie in the position and the length of the plateau corresponding to saturation of the amino groups.

To account for the dye which is adsorbed in excess of the free amino groups, Peters [2] suggested that combination between the dye and the peptide (–CO·NH–) groups occurs, either by adsorption of a hydrogen ion to produce a charged site, the dye ion following to maintain electrical neutrality, or by adsorption of the uncharged dye acid. The lack of a maximum in the adsorption is then explained by the large number of peptide groups present.

Adsorption of undissociated acids appears the more feasible, and has been postulated by Harris *et al.*[4] to account for the adsorption of some organic acids by wool. The idea receives some confirmation from the work of Elöd and Fröhlich [3], who demonstrated that adsorption is greater the weaker the acid, since the latter will be associated to a greater extent. The attachment of the dye to the fibre will then occur by means of hydrogen bonds or dipolar interaction between suitable groups in the dye molecule and the peptide groups in the fibre chains. If this is true, it would seem possible to adsorb more dye on nylon than corresponds to "amine-group" dyeing under conditions of high salt and dye concentrations. This is confirmed by the work of Fern [5] using Naphthalene Orange G under neutral conditions.

Recently, however, some doubt has been thrown on this view of the "overdyeing" phenomenon, and it has been suggested that the dye adsorbed in excess of the amino group content of the fibre at low pH is due to hydrolysis of the main chains, which produces sites suitable for additional adsorption of dye, and involves a corresponding amount of degradation in the material. Such possibilities were dismissed by earlier workers [2,6,7] since no degradation was detected when nylon was treated with mineral acids under comparable conditions of acidity and temperature. Even boiling for 2 hr. in a dyebath containing Solacet Fast Blue 2B at a pH of 1·0 was shown by Carlene, Fern, and Vickerstaff [8] to produce only a small change in fluidity of their nylon, and they concluded that no serious hydrolysis had occurred.

Contrary to these results are those of Remington and Gladding [9], who dyed nylon at 80°c. at low pH and showed considerable hydrolysis by means of changes in fluidity before and after dyeing and by the shape of the rate-of-dyeing curve. There is a rapid adsorption of dye in the initial stages, followed by a slow but steady increase, which apparently does not reach a maximum. They showed quite conclusively that the second part of the curve arises from hydrolysis of the main chains of the nylon. Hydrolysis with mineral acids has recently been demonstrated by Elöd and Fröhlich [3], who treated nylon at 90°c. with 0·05 N. hydrochloric acid. These authors showed by viscosity measurements appreciable changes in the molecular weight of the polymer even after 6 hr., and more substantial effects after 48 hr., the drop in molecular weight being from 12,500 to 7,300. On the views of Remington and Gladding, who consider the process to be catalysed by the presence of dye, the hydrolysis of dyed fibres would be quicker than that shown by Elöd and Fröhlich with hydrochloric acid.

Thus, two explanations of the "overdyeing" have been developed which, at first sight, appear to be incompatible. It seemed to the present authors that the truth might lie in a combination of these theories as suggested by Palmer [10], and that the process might be initial adsorption of dye on the peptide groups followed by a hydrolysis which would be fairly slow. If this is so, then the different results obtained by different workers are capable of a rational explanation. At 60°c., the temperature employed by Peters, the hydrolysis may have been too slow to be noticeable, whereas it would be detectable at the higher temperatures used by Remington and Gladding and by Elöd and Fröhlich. The results of Carlene, Fern, and Vickerstaff can be accounted for by the fact that their dyeings were carried out for short periods (about 2 hr.), during which only a small amount of hydrolysis could take place.

The occurrence of two such mechanisms of dyeing has been postulated by the American workers, who visualise the dye being adsorbed on peptide groups, the concentration of hydrogen ions in the fibres being thereby increased with the result that the rate of hydrolysis is increased.

The present work was undertaken to throw further light on the validity of this viewpoint.

EXPERIMENTAL

The experiments fall into two groups—

(*a*) Those designed to show that hydrolysis cannot entirely account for the dye uptake at low pH. In these experiments the fibre was dyed, stripped, and redyed to estimate the number of free amino end-groups.

(*b*) Viscosity measurements designed to follow molecular weight changes during the dyeing.

The materials used in these experiments were—

(i) Micro dull nylon yarn, 15 denier, containing 0·035 milliequivalent of amino end-groups per gram of fibre, as estimated by conductometric titration in 90% phenol–water with alcoholic hydrochloric acid. The nylon was scoured for 20 min. in a bath containing soap (3 g./litre) and ammonia (1 c.c. per litre) at 60°c., and then thoroughly rinsed in distilled water.

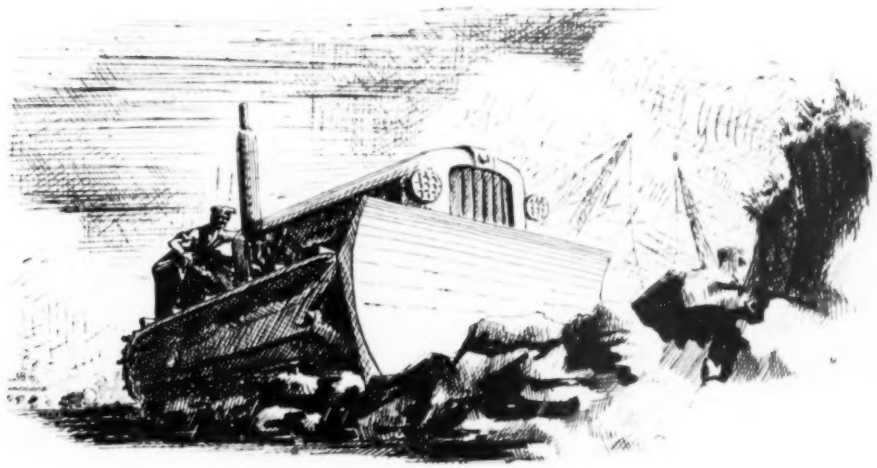
(ii) Commercial brands of dye were used, the dyebaths being made up at the following strengths—

Lissamine Fast Yellow 2G 125	8 g./litre
Naphthalene Orange G 125 ...	8 g./litre
Solway Blue B 130	6 g./litre

The pH of the dyebath was adjusted with sulphuric acid (A.R. quality).

For colorimetric estimations, samples of the purified dyes were employed to give standard optical densities.

Rate-of-dyeing curves of nylon were determined at low pH, the resulting dyeings being stripped and redyed at high pH, under conditions in which only amino-group dyeing occurs. The experiments were



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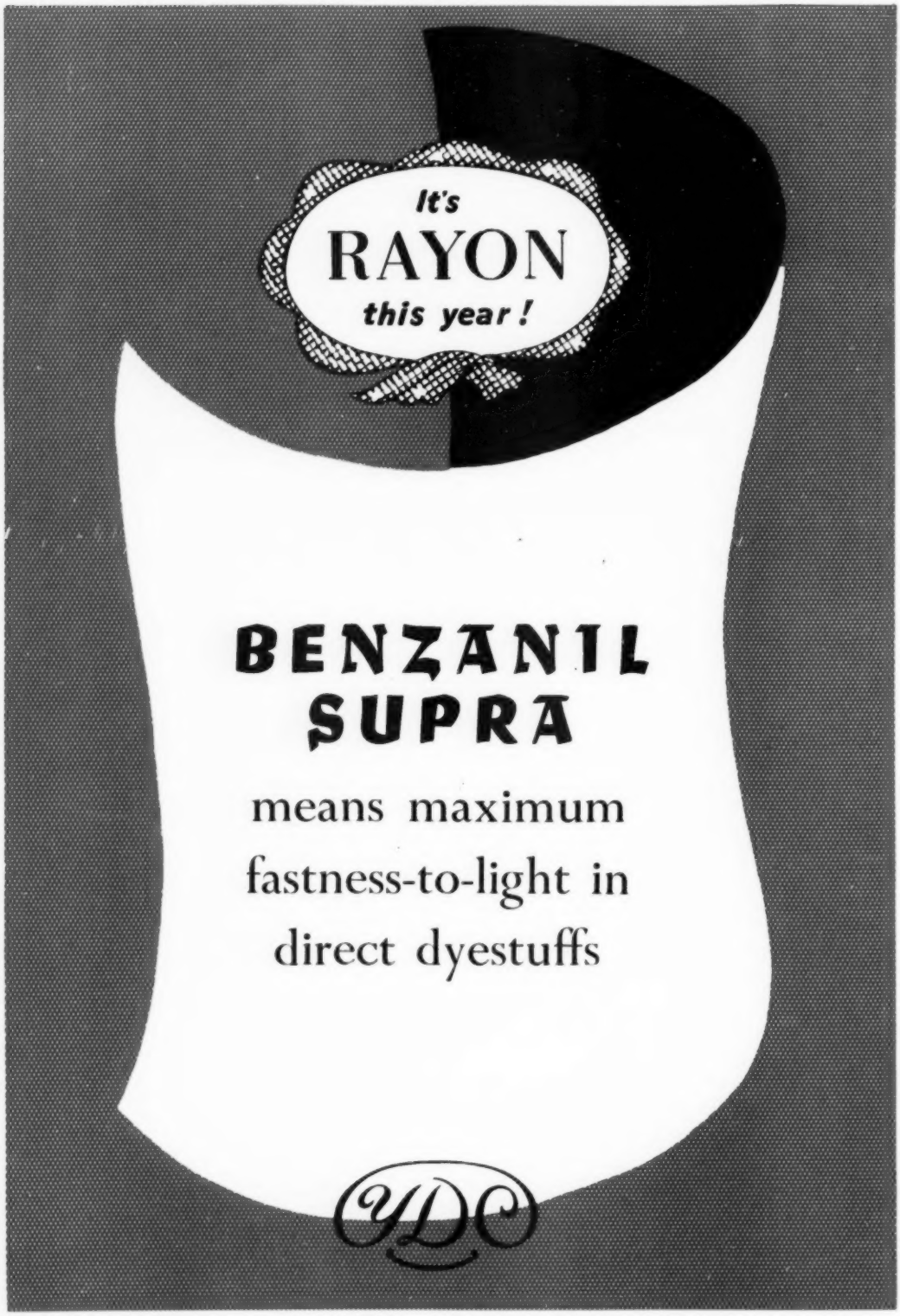

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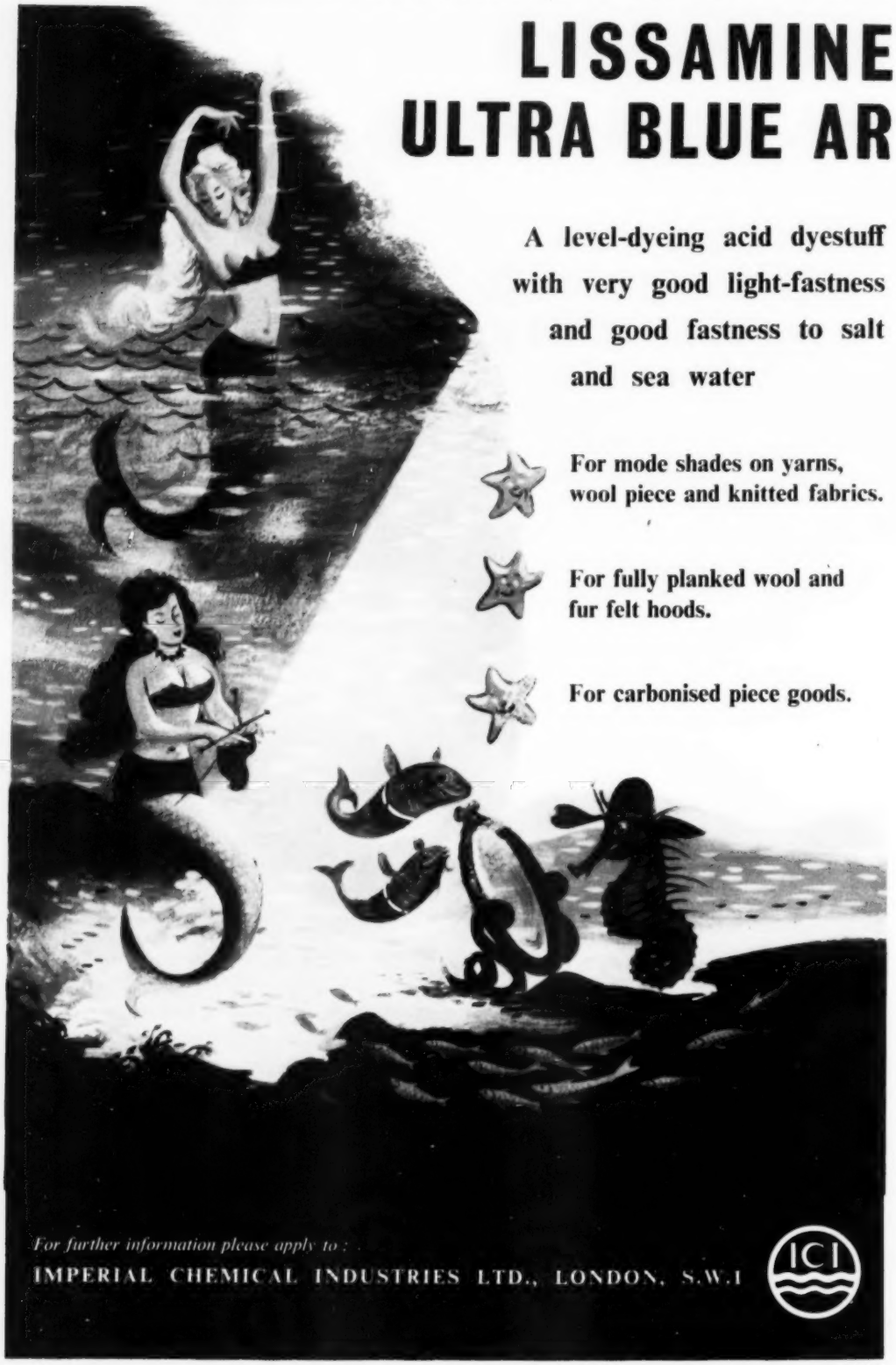

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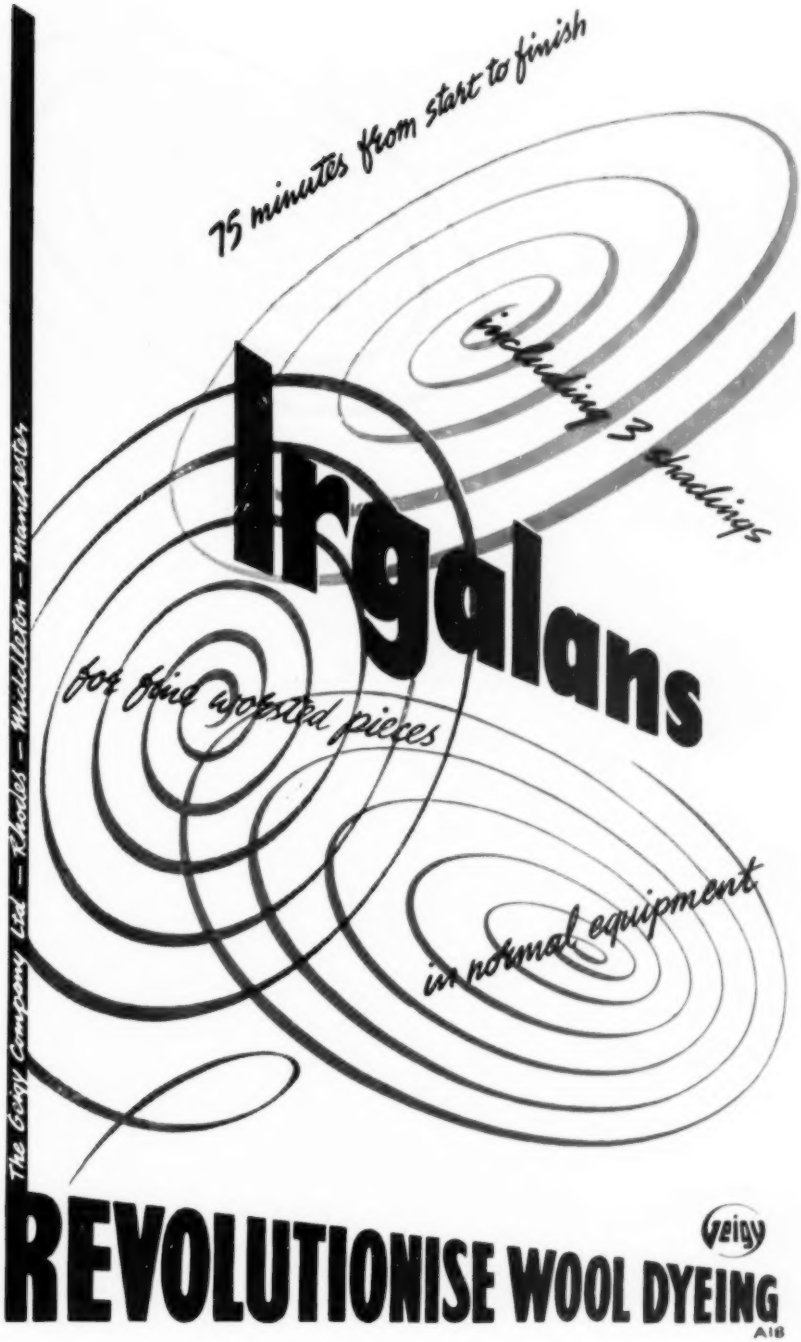
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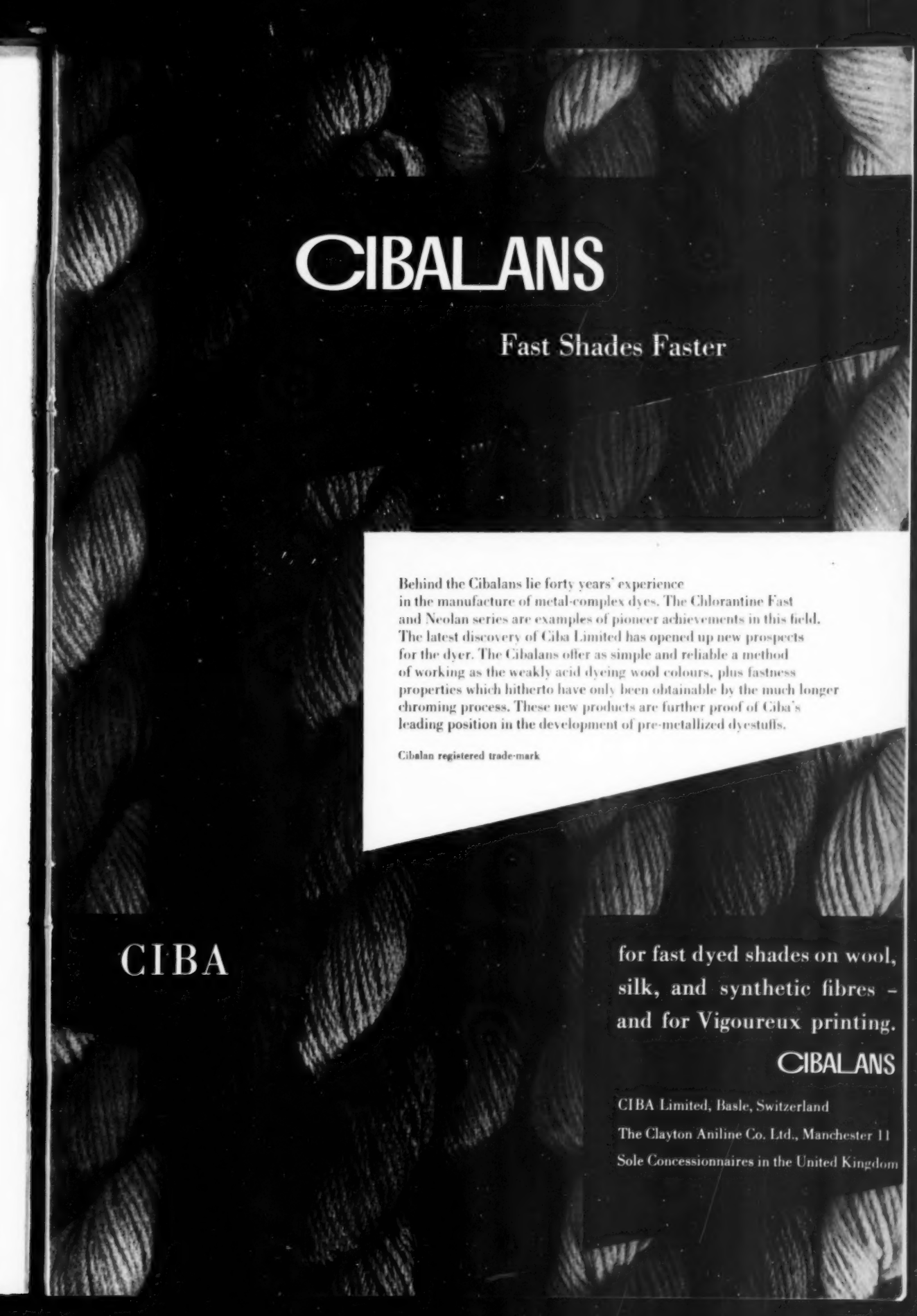
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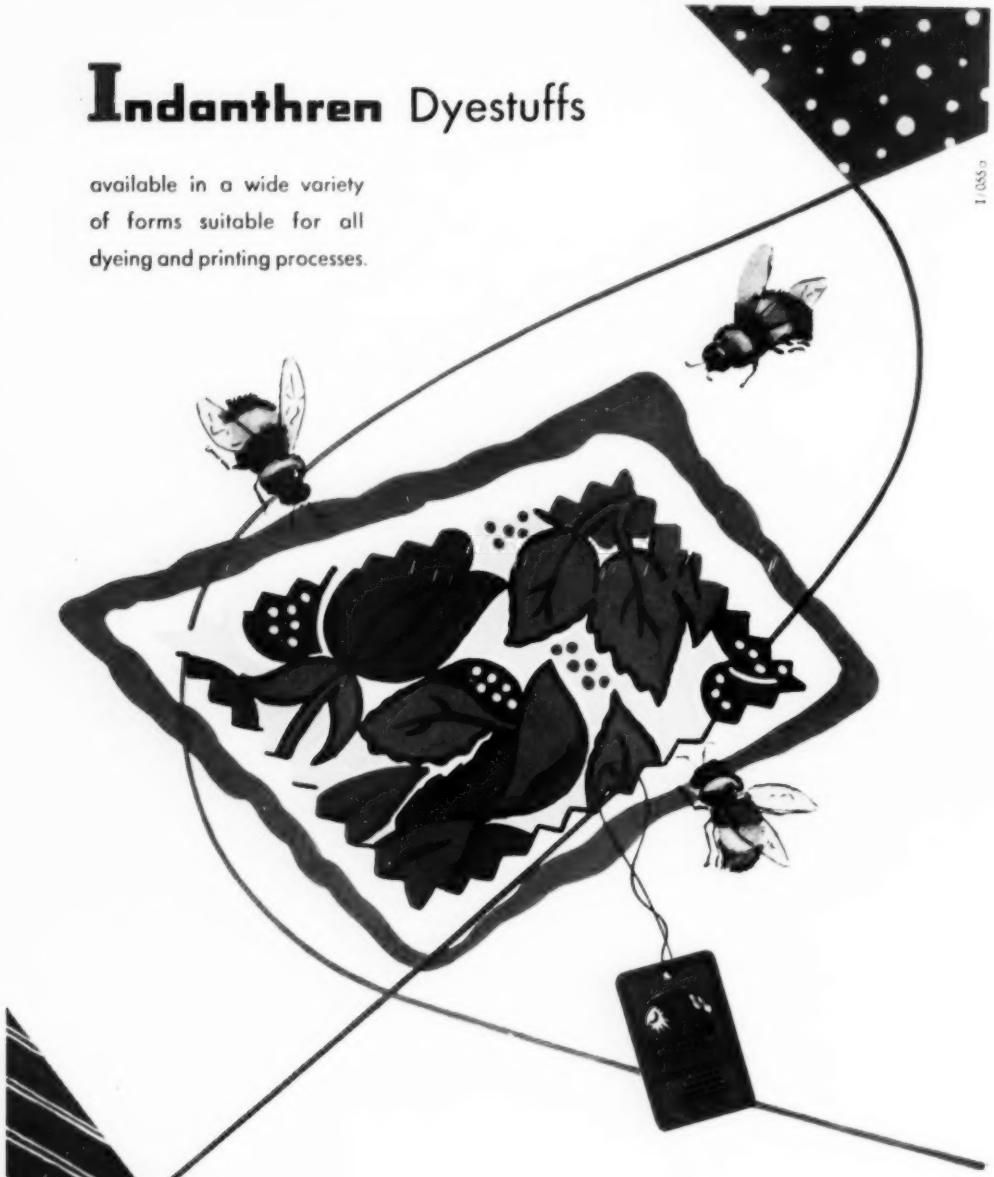

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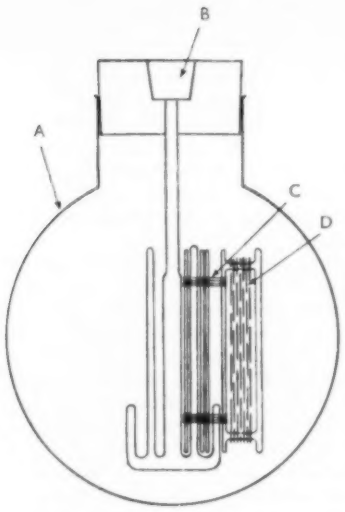

FIG. 1

carried out in the apparatus shown in Fig. 1. The dyeing vessel was a 3-litre bolthead flask with a rubber bung through which passed a Quickfit (Quickfit & Quartz) airtight-gland stirrer. Passing through the gland was a glass rod reaching nearly to the bottom of the flask, and fused symmetrically around the end of the rod were six glass frame holders, the longer arms of which projected out of the liquid. The nylon was wound on glass frames which were attached to the arms of the stirrer.

Each hank weighed 1·5 g. and the frames were of such a size as to carry two hanks. The stirrer revolved thirty times a minute.

The dyeing procedure was as follows—

Twelve hanks were put on the frames which were suspended in the vessel, and the dye solution, adjusted to the requisite pH (ca. 1·5) and preheated to 60° C., was run in. After a period of time had elapsed, the stirrer was stopped, a frame removed, and a hank cut off. The frame was returned to the dyeing vessel and the stirring recommenced, the whole operation taking only one minute. The hank was washed in cold distilled water and divided into two parts. One of these was conditioned at 65% R.H. prior to being dissolved in freshly distilled *o*-chlorophenol and the dye concentration estimated from the optical density of the resulting solution. The other part was placed in a decinormal solution of ammonium hydroxide at 60°C. to neutralise the acid and strip the dye from the fibre. By this means complete removal of the dye from the fibre was not possible even after six or seven changes of the desorbing liquor. The residual amount of dye was considerably less than that required to saturate the amino groups which were present in the undyed fibre. Since this was adequate for the purpose of demonstrating the increase in number of amine groups present in the overdyed nylon, no attempt was made to remove all the remaining dye from the fibre. The stripped nylon was redyed in a solution of the same dye concentration as the first dyeing but at pH 5·0 for 72 hr. Some of the results are given in Table I for Lissamine Fast Yellow 2G.

TABLE I

Effect of Dyeing at Low pH on Amine Content of Nylon Yarn

(Lissamine Fast Yellow 2G. Final pH = 1·48)

Time of Dyeing (hr.)	Dye adsorbed (m-equiv./g. fibre)		
	Original Dyeing (i)	Redyeing (= NH_2)	Excess in (i) over NH_2 Content
2	0·073	0·040	0·033
17·5	0·112	0·049	0·063
19	0·117	0·047	0·070
20·5	0·116	0·047	0·069
24	0·117	0·049	0·068
26	0·121	0·052	0·069
42	0·128	0·055	0·073
43	0·125	0·052	0·073
45	0·127	0·059	0·068
46	0·130	0·063	0·067
49	0·130	0·057	0·073
50	0·127	0·063	0·064
66·5	0·138	0·070	0·068
68·5	0·139	0·068	0·071
72·5	0·138	0·072	0·066
72·5	0·140	0·072	0·068
74·5	0·152	0·069	0·093
91	0·143	0·076	0·067
96·5	0·146	0·076	0·070
122·5	0·166	0·081	0·085
139	0·170	0·089	0·081
142	0·170	0·093	0·077
162·5	0·176	0·099	0·077
162·5	0·177	0·097	0·080

Three dyes in all were used — Lissamine Fast Yellow 2G, Naphthalene Orange G, and Solway Blue B. Since the results on all three were similar only those for Lissamine Fast Yellow 2G are given here (Fig. 2). Curve (*a*) represents the rate of dyeing at low pH, and shows the characteristics

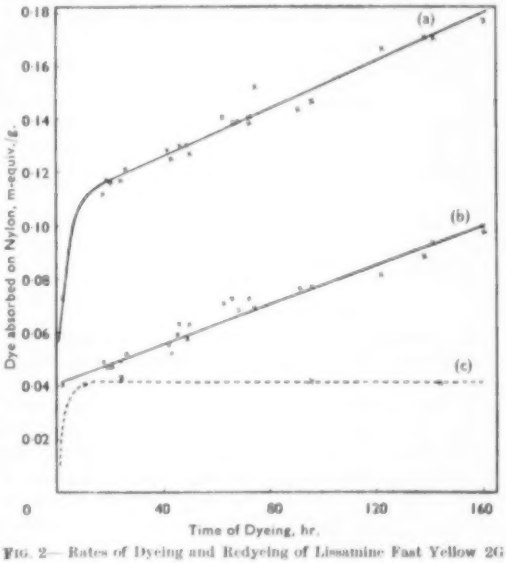

FIG. 2— Rates of Dyeing and Redyeing of Lissamine Fast Yellow 2G

noted by Remington and Gladding. Curve (*c*) represents the rate of dyeing at high pH ("amino group" adsorption), whilst curve (*b*) gives the amount of dye adsorbed by the fibre after it had been dyed at low pH, stripped, and redyed under the same conditions as for curve (*c*). The points of curve (*b*) correspond, therefore, to those on (*a*), and show how the total number of free amino groups present in the fibre increases with time of dyeing from baths of low pH.

With all three dyes, curve (*b*) was found to be considerably below (*a*), and intersected the ordinate at a point which corresponded to the maximum value of the adsorption of dye under conditions of high pH, curve (*c*).

Viscometric measurements were carried out on the overdyed samples of nylon in a 90% phenol–water mixture as solvent, since *o*-chlorophenol and 90% formic acid gave erratic results. The measurements were made at 25 ± 0·01°C. in a modified Ostwald viscometer (No. 1) fitted with an enlarged receiving bulb so that the concentration could be altered by dilution with pure solvent. The times of flow were always greater than ten minutes, and so fulfilled the requirements for maximum accuracy recommended by Hartley [11]. At least three readings of each flow time were made, and half an hour was allowed to elapse after each dilution.

The results are given in Table II. The molecular weights have been calculated from the intrinsic viscosity η_0 by means of Mark's formula—

$$\eta_0 = KM^{\alpha}$$

where M is the molecular weight. Appropriate values of K and α have been taken to be $1{\cdot}56 \times 10^{-3}$ and 0·66 respectively, from work carried out in the laboratories of Messrs. British Nylon Spinners Ltd. [12]. It can be seen from the figures that a steady decrease in molecular weight occurs with time, showing that hydrolysis had taken place during the dyeing period.

Table II

Molecular Weights of Overdyed Nylon

Dyeing Time (hr.)	Dye on Fibre (m-equiv./g.)	η_0	M
	Lissamine Fast Yellow 2G		
19	0·117	0·674	9800
72·5	0·138	0·572	7670
162·5	0·176	0·423	4850
	Naphthalene Orange G		
17·5	0·180	0·643	9180
72·5	0·205	0·507	6400
162·5	0·242	0·380	4130

Discussion and Conclusions

In studying these results it is convenient to summarise the evidence which points to hydrolysis of the fibre. The rates of dyeing at low pH (curve (*a*), Fig. 2) show no sign of reaching a maximum even after 120 hr., indicating that new sites for adsorption of dye are being created as dyeing proceeds. This steady increase in the uptake of dye is paralleled by the dyeings carried out on the stripped fibre (curve (*b*)), which show a corresponding increase in the number of amine groups liberated during the dyeing. The viscometric results also confirm the fibre degradation and show a steady decrease with time, although the molecular weights decrease more rapidly than the overdyeing results would indicate. To complete this comparison the molecular weights calculated from the overdyeing experiments by assuming that it is proportional to the number of end groups are given in Fig. 3 and 4. The curves so obtained run parallel in the later stages of the hydrolysis but not in the initial stages.

A direct comparison between the molecular weights deduced from the two methods is difficult for the following reasons. The viscometric technique gives an average value which is weighted to a larger extent than that based on the free amine group determination. It is not certain that there is an accurate 1 : 1 correspondence between the number of free amine groups and molecular weight. Finally, although the solvent employed here is the best available, the high concentrations of dye used in these experiments may influence the viscometric results by causing changes in the configuration of the dissolved nylon molecules. The results are nevertheless adequate to show that substantial hydrolysis had occurred in the overdyeing treatment.

However, it is clear that hydrolysis is not the complete explanation of the adsorption in excess of

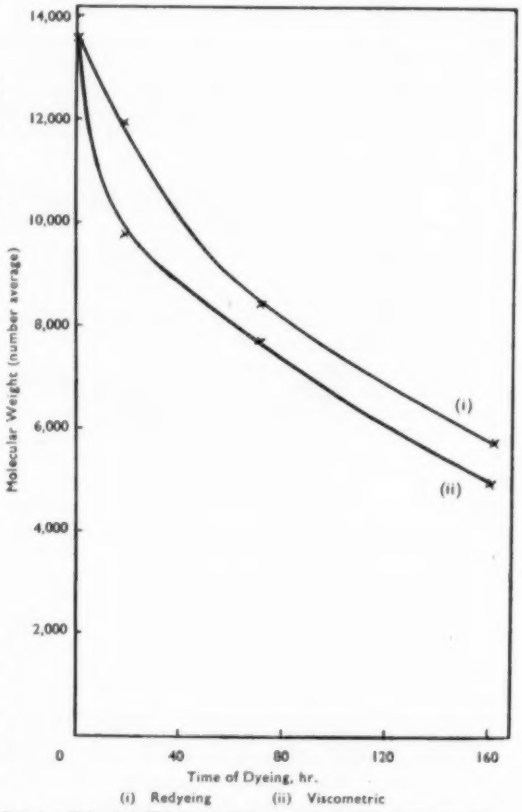

Fig. 3— Molecular Weight of Nylon overdyed with Lissamine Fast Yellow 2G

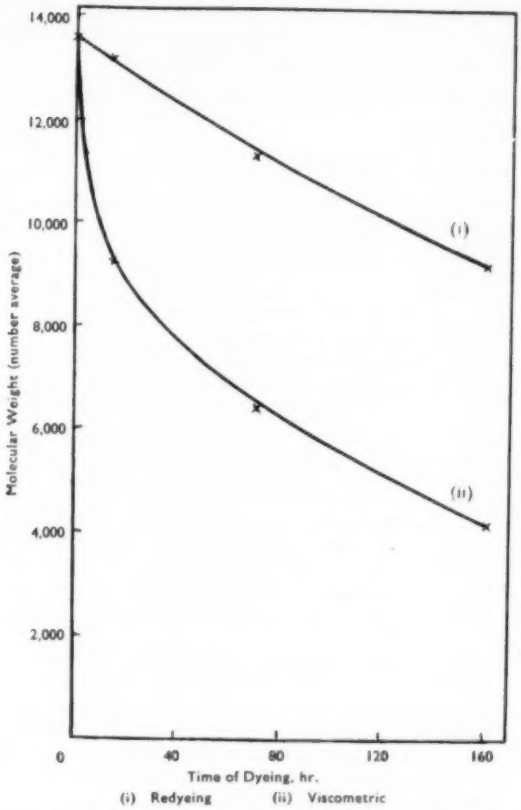

Fig. 4—Molecular Weights of Nylon overdyed with Naphthalene Orange G

the free amine groups initially present in the fibre. The amine group content of the overdyed fibre given by curve (*b*) (Fig. 2) is considerably smaller than the amount of dye which the fibre is capable of adsorbing from baths at low pH. Curve (*b*) does, however, run parallel to curve (*a*), which confirms the fact that the steady increase in dye uptake at low pH values is due to hydrolysis. A comparison of curves (*a*) and (*b*) shows that it is possible for the fibre to adsorb approx. 0·075 milliequivalent of dye in excess of that which can be accounted for by simple attachment to the amino groups. To explain this, recourse must be made to previously discussed hypotheses that the dye is adsorbed on the peptide groups in the fibre. The results therefore confirm the view that there are two mechanisms at work in the dyeing process—an initial high uptake of dye by adsorption on peptide groups, followed by a steady hydrolysis which is represented by curve (*b*)

Finally, the hydrolysis could occur at two places in the nylon molecule, namely at the end of the chain to remove an acyl residue or at one of the peptide groups along the chain. In the former case the molecular weight calculated from dye adsorption would decrease, but not that found viscometrically. In the second case both would decrease. Since the experimental results are of the latter type, it must be concluded that hydrolysis is, in the main, occurring at the peptide groups in the main chain.

Summarising then, the experiments described here show that acid dyeing of nylon at low pH occurs by adsorption of the dye on peptide groups. The presence of the dye catalyses hydrolysis of the fibre, thereby causing the dye uptake to increase indefinitely, with a corresponding decrease in the molecular weight of the material.

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Studies in the Fundamental Processes of Textile Printing
VI—The Transfer of Disperse and Water-soluble Dyes to Nylon during Steaming

E. H. DARUWALLA and H. A. TURNER

The work reported in Part V of this series [1] has been extended to include a study of the transfer of disperse, soluble acetate-rayon, and acid dyes to nylon. In general, there is a close resemblance between the dyeing behaviour of each class of dye and the corresponding transfer behaviour during steaming. This is demonstrated by a comparison of the present experimental results with those which have been obtained in quantitative studies of nylon dyeing by other workers.

Introduction

GENERAL

The nylon (superpolyamide) group of fibre-substances are particularly interesting because, while they resemble the polyester fibres in possessing a high affinity for disperse dyes [2], they differ in behaving as characteristically basic fibres, and are thus able to fix soluble anionic dyes from neutral or acid baths [3–5].

Earlier experiments have already confirmed practical experience by showing that the transfer of water-soluble acid and acetate-rayon dyes to cellulose acetate during steaming is not very great in the absence of suitable fibre-swelling or dye-dispersing agents [1]. With nylon, as might be expected, the uptake of dyes of these classes can be very large, and it can be controlled primarily by adjusting the concentrations of hydrogen and other readily diffusible ions in the thickener phase. The experimental technique used in these studies [6, 1] to investigate the transfer of dye from thickener to fibre-substance during steaming requires that this fibre-substance shall be in the form of a regular film. Up to the present, therefore, the experimental systems have had, of necessity, to represent the manufactured fibres rather than the natural ones. It has been difficult to find a system in which the behaviour of representative fibrous proteins might be investigated, for the preparation of suitable films of these materials, in a form that would withstand prolonged steaming, has not yet been accomplished. Until they can be made, nylon does at least, represent a basic fibre of relatively simple constitution, and, being thermoplastic, allows films to be made without great difficulty. There have been a number of practical investigations to find the best conditions for colouring the textile nylons by dyeing and printing, and there are also some careful laboratory studies which can provide comparative data and hypotheses during the discussion of the present results.

SUMMARY OF RESULTS

(1) The transfer of selected disperse dyes, viz. Dispersol Fast Scarlet B and its higher homologue, Disperse Dye A*, both pure preparations, from starch to 66 nylon during steaming, follows the general course of the corresponding transfer to cellulose acetate in that —

(*a*) The initial rate of exhaustion is high.

(*b*) Equilibrium exhaustion values are high (90%) and are independent of the initial dye concentration in the starch film, up to the point where saturation of the fibre-substance occurs.

(*c*) From (*b*) above, it follows that the constant partition of dye between fibre-substance and thickener, taken as characteristic of the solid-solution theory of dyeing for cellulose acetate, is observed also in the transfer of disperse dyes to nylon.

(*d*) The overall rate of exhaustion increases as the initial concentration of dye in the thickener is made smaller.

(*e*) The rate of dye transfer and the value for equilibrium exhaustion are almost unaffected by changes in the pH of the system.

(2) Unlike cellulose acetate, the transfer of soluble acetate-rayon dyes (Solacet Fast Scarlet B and Soluble Dye A*, both pure) is rapid, and the value for equilibrium exhaustion is high. Transfer to nylon increases as the pH of the system is diminished, and, at a given pH, is made less by the addition of sodium chloride.

(3) An equation may be found to fit the experimental rate-of-transfer curves for both disperse and soluble dyes for acetate rayon. From these, the calculated velocity coefficients show that—

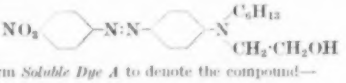
(*a*) As the initial concentration of the dye in the thickener is made larger, the rate of transfer of both disperse and soluble dyes is diminished.

(*b*) With standard experimental conditions, the rate of migration of Dispersol Fast Scarlet B ($R{\cdot}C_2H_5$) is greater than that of its homologue, Disperse Dye A ($R{\cdot}C_6H_{13}$); for the corresponding sodium sulphuric esters, Solacet Fast Scarlet B and Soluble Dye A, the order is reversed.

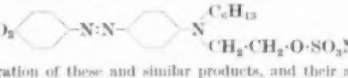
(*c*) The rate of transfer for each of the disperse dyes named in (*b*) above is less than that for the corresponding soluble derivative.

(4) Together with the soluble dyes already mentioned, the acid group was represented by three

* As in the previous paper [1], the term *Disperse Dye A* is used to denote the compound —



and the term *Soluble Dye A* to denote the compound—



The preparation of these and similar products, and their application in the dyeing of textile fibres are described in *BP* 575,342[7].

dyes obtained by introducing different numbers of sulpho (SO_3Na) groups into the naphthylazo-β-naphthol molecule, viz. Naphthalene Red J (one), Naphthalene Red EA (two), and Naphthalene Scarlet 4R (three). Following are the most important results—

(*a*) An increase in the number of sulphonic acid groups in the dye molecule causes a reduction both in the rate of transfer to the nylon and in the proportion of dye transferred at equilibrium.

(*b*) A decrease in the pH of the system causes an increase in the rate and the extent of exhaustion for all the acid dyes. At pH 2·3, addition of sodium chloride to the thickener decreases the proportion of dye transferred at equilibrium; at pH 6·8, similar additions have no effect.

(*c*) At pH 4, in systems where there is an excess of acid dye in the thickener phase beyond that which corresponds to saturation of the nylon, the number of equivalents of dye in the nylon corresponds approximately to the number of free amino groups in the fibre-substance. The closest approach to strict equivalence is found with Naphthalene Red EA. With Naphthalene Red J, the adsorption of dye exceeds strict equivalence; with Naphthalene Scarlet 4R it is less.

(*d*) On steaming a film of nylon containing a high proportion of Naphthalene Red EA in contact with a plain starch film containing sodium chloride, dye is desorbed readily from the nylon until the proportion remaining in the fibre-substance is slightly less than equivalent to the free amino groups in the nylon.

(5) Reduction in the proportion of the free amino groups in the nylon by acetylation causes—

(*a*) Practically no change either in the rate of transfer or in the proportion of dye transferred at equilibrium for Dispersol Fast Scarlet B.

(*b*) A reduction in the exhaustion at equilibrium for both soluble acetate-rayon and acid dyes, the reduction being different for different dyes.

Experimental

MATERIALS

Dyes

The preparation and purification of the disperse and the soluble dyes for acetate rayon has been fully discussed in Part V of this series [1]. For the present experiments, the disperse dyes were ground in distilled water for 20 hr. before use.

NAPHTHALENE RED J (ICI, *C.I.* 176)—The commercial brand was crystallised four times from boiling distilled water. This operation was initially difficult because the crystals were very fine, and the difficulty became greater with each crystallisation. After filtration from the last, the dye was dried and taken as sufficiently pure and free from electrolyte to be suitable for the experiments.

NAPHTHALENE RED EA (ICI, *C.I.* 182)—This was crystallised four times from hot aqueous–alcoholic solution. At the last two stages, the optical density in standard solution (20% aqueous pyridine) remained unchanged.

NAPHTHALENE SCARLET 4R (ICI, *C.I.* 185)—This was purified and the purity checked in the same manner as for Naphthalene Red EA.

Nylon Film

Film from two sources was used. Each kind was made from 66 nylon, the end-amino-group contents being ICI 0·033, Du Pont 0·042 gram-equivalent per kilogram total dry weight. The sheets of ICI material were made by pressing a calculated weight of the nylon salt in a sheeting press, the platens of which were maintained thermostatically at a temperature just above the fusing point of the material. Aluminium foil was interposed between the platens and the molten salt. After cooling, the foil was stripped from the pressed film. No flux, or lubricant to assist sheeting, was present, as it might later have interfered with dye transfer. The du Pont film was obtained ready-made, appearing, from the stress marks on the surface, to have been extruded, or formed by a continuous casting method. Neither source yielded film of completely uniform thickness. Each supply was therefore cut into small strips, which were sorted into groups of similar thickness. Only the groups containing film 0·0040–0·0045 in. thick and 0·0045–0·0050 in. thick were used, samples for any one set of experiments being all taken from the same group. The strips were soaked in several changes of distilled water, and were kept in distilled water until needed.

Acetylation of Nylon

This was treated in film form by the method of Peters [3]. Dry nylon film (10 g.) in strips was heated for 48 hr. at 80°c., under reflux, with 60 g. acetic anhydride in 180 ml. benzene. The product was washed with benzene, acetone, and water and dried, and the end-amino-group content was determined by the method of Waltz and Taylor [8].

PROCEDURE

Preparation of Dye-containing Starch Film

It was found that dry starch films could readily be stuck to nylon, in contrast to the behaviour of cellulose acetate, and could be retained in contact during prolonged steaming. Accordingly, the normal method of making starch films, drying them, and cutting them, as described in previous papers [6,9], was adopted without essential modification.

Attachment of Starch Films to Nylon

The dye-containing starch films, cut into discs of standard area, were conditioned over water for a short time and then squeezed firmly, the inclusion of air bubbles being avoided, into contact with water-soaked nylon film from which excess water had been removed with filter paper.

Steaming

The procedure has already been fully described [6].

Estimation of Dye in Each Phase

After steaming, the starch and the nylon layers were separated by hand. Dye in the former was extracted with a known volume of 20% aqueous pyridine solution, and the dye present estimated colorimetrically. Dye in the nylon was determined by dissolution of the whole film, including dye, in a known volume of 90% aqueous formic acid solution, followed by colorimetric estimation.

Determination of pH

For experiments on dye transfer at different pH values, the pH of the printing paste was first adjusted by the addition of suitable proportions of Sørensen borate–acid and borate–alkali buffer mixtures. The pH at room temperature was then determined by means of the glass electrode. It was known that there would be a change in pH after the starch had been dried, and again when it had been in contact with nylon in the steaming apparatus. To determine the approximate magnitude of this change, control experiments were made with buffered starch films. In these, the dye had been left out but a suitable indicator had been added. The colour of this was observed when the control sample was in the steam. It was found that the change in pH, from that in the original paste, was always less than one unit. Since the effects of comparatively large differences in pH were being examined, it was felt that this disparity could be accepted.

Experimental Results

TRANSFER OF DISPERSE DYES

Rate-of-transfer curves for the migration of the dye from the starch to the nylon during steaming, representing different proportions of total dye in the system, are set out in Fig. 1 for Dispersol Fast Scarlet B, and in Fig. 2 for Disperse Dye A (one concentration only). These, and other results, have been used to construct the adsorption isotherm for Dispersol Fast Scarlet B (Fig. 3). As with the transfer of the same dyes to cellulose acetate [1], it is found that— (i) transfer is initially rapid, exhaustion being relatively faster the less the proportion of dye in the system; (ii) the final exhaustion is high, 96–97% for Dispersol Fast Scarlet B, 88% for Disperse Dye A, and is independent of the proportion of dye in the system until saturation of the nylon with dye has been attained. More precise data concerning the rate of dyeing are given later. At this stage, attention is called to Fig. 9, from which it is seen that the transfer of disperse dye is almost independent of the pH of the system.

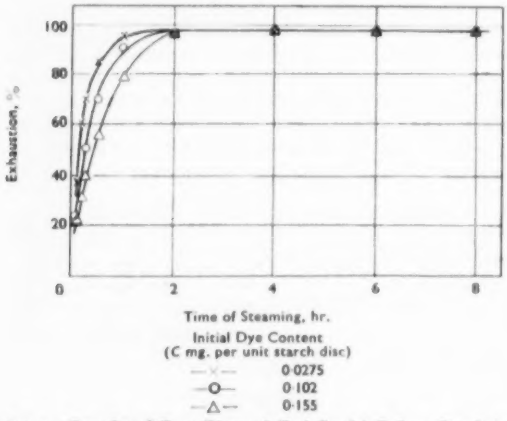


FIG. 1—Transfer of Pure Dispersol Fast Scarlet B from Starch to Nylon during Steaming

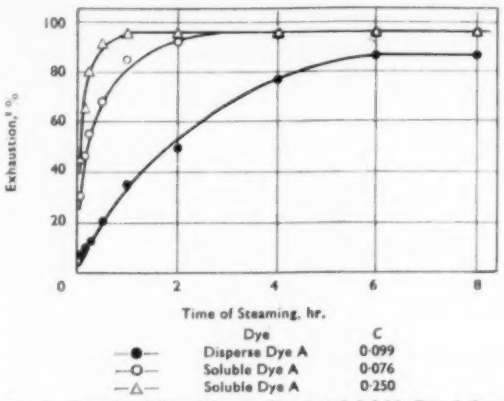


FIG. 2—Transfer of Pure Disperse Dye A and Soluble Dye A from Starch to Nylon during Steaming

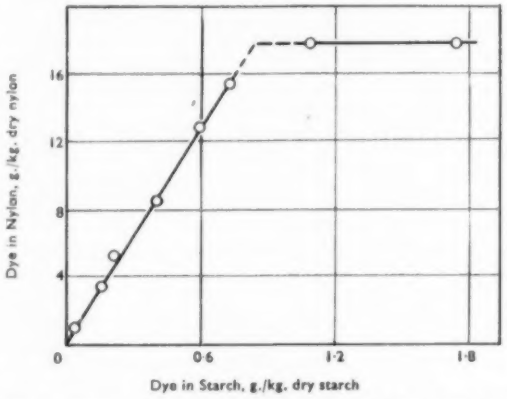


FIG. 3—Isotherm for Distribution of Pure Dispersol Fast Scarlet B between Starch and Nylon at Equilibrium

TRANSFER OF SOLUBLE DYES FOR ACETATE RAYON

In these experiments, the transfer of Solacet Fast Scarlet B (Fig. 4) and of Soluble Dye A (Fig. 2) occurred in the absence of a buffer. Unlike the corresponding transfer to cellulose acetate [1], the exhaustion is very high, reaching 98% for the first dye and 95% for the second. The greater part of this transfer is accomplished in the first hour of steaming, and, with the lower initial concentration, equilibrium conditions are soon established. With the higher concentrations of Solacet Fast Scarlet B, the rate of exhaustion is less and true equilibrium has not been reached after 8 hr. It appears that, could the time of steaming be prolonged until equilibrium has been established, the exhaustion would be practically independent of the initial concentration of dye in the system. As these are

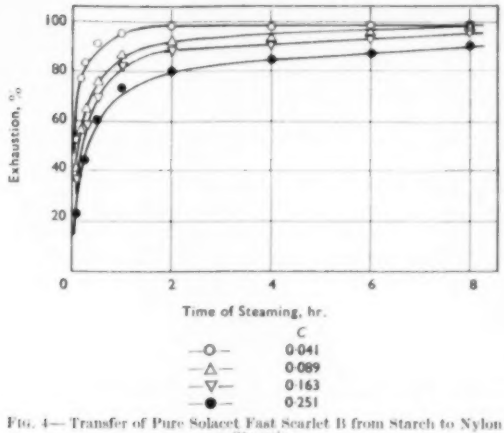


FIG. 4—Transfer of Pure Solacet Fast Scarlet B from Starch to Nylon on Steaming

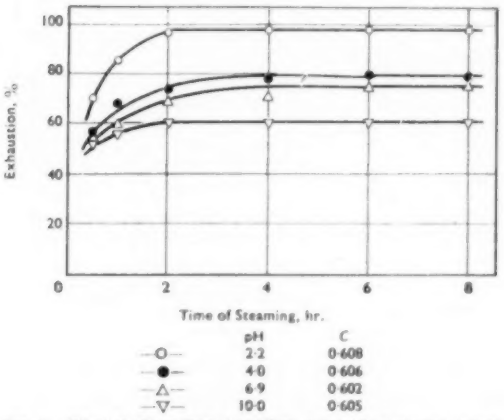


FIG. 5—Effect of pH on Transfer of Pure Solacet Fast Scarlet B from Starch to Nylon during Steaming

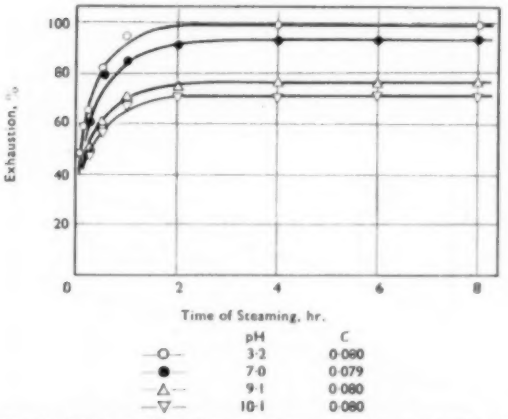


FIG. 6—Effect of pH at Low Initial Concentration on Transfer of Pure Solacet Fast Scarlet B from Starch to Nylon during Steaming

essentially acid dyes, it is not surprising to find that the exhaustion rises when the pH of the system is lowered (Fig. 5 and 6), but even at high pH values the exhaustion is appreciable.

TRANSFER OF ACID DYES

The first series of experiments was carried out with Naphthalene Red EA (two sulphonic acid groups). This dye, from the results of Meitner [10] and of Pomfret [11], is shown to be readily soluble, with a small particle radius at room temperatures, exhausting in printing experiments on cellulose to a moderate extent only, and passing early through the characteristic maximum in the exhaustion, similar to that found by Patel and Turner [12] for Naphthalene Scarlet R. The effect of changing the pH in steps of 2–3 units from 10 to 2 is shown in Fig. 7. As would be expected, exhaustion is more rapid and goes nearer to completion at the lower pH values, but even at pH 10 there is an ultimate

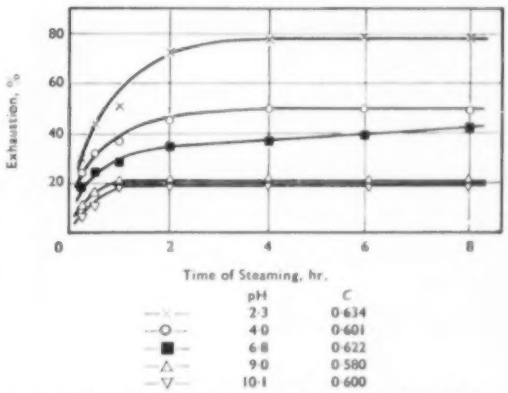


FIG. 7—Effect of pH on Transfer of Naphthalene Red EA from Starch to Nylon during Steaming

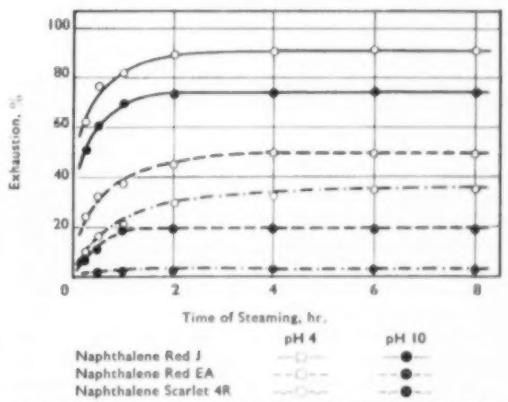


FIG. 8—Effect of pH on Transfer of Acid Dyes from Starch to Nylon on Steaming

value of about 18%, so that not all the dye transfer is due to the presence of amino groups in the nylon. Fig. 8 gives the exhaustion of each of the three acid dyes at the two pH values of 4 and 10. Exhaustion of the monosulphonated dye (Naphthalene Red J) is very high at pH 4, but it is also higher than the exhaustion of any of the other acid dyes, including the soluble dyes for acetate rayon, at pH 10. As Naphthalene Red J has the lowest proportion of water-solubilising groups in the molecule, it would appear at the high pH value to be retained by the

nylon, in part, by the same mechanism as that which causes the adsorption and retention of disperse dye molecules, i.e. by forces other than electrovalencies. As the basicity of the acid dyes increases, through Red EA to Scarlet 4R, with the simultaneous increase in the water-solubility and in the probability of attachment to the fibre-substance through electrovalencies, the exhaustion at a given pH diminishes and the change in the exhaustion resulting from a given change in the pH increases (Fig. 8).

A comparison of the effect of pH on the equilibrium exhaustion of disperse, acid, and soluble dyes for acetate rayon is given in Fig. 9.

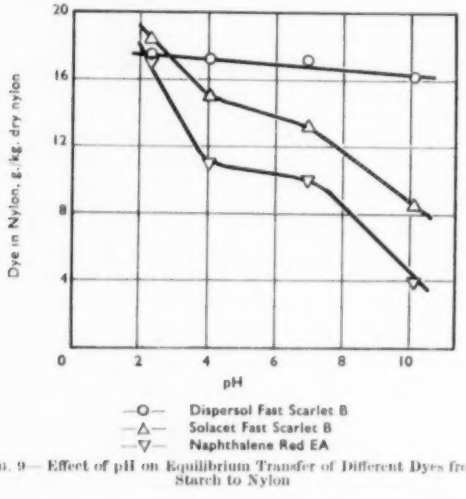


FIG. 9—Effect of pH on Equilibrium Transfer of Different Dyes from Starch to Nylon

EFFECT OF TERMINAL AMINO GROUPS

Other workers have attempted to assess the relative importance of electrovalencies and other types of chemical bond in the attachment of acid dyes to nylon by dyeing nylon in which the terminal free amino groups are protected by acetylation [3,13]. Acetamido groups remain unionised until the dyebath is strongly acid. Correspondingly, the transfer of a variety of acid dyes to nylon sheet by steaming has been determined for the normal material (0·042 g.-equiv. NH_2 per kg.) and compared with the results for partly acetylated material (0·0041 g.-equiv. per kg.). In Fig. 10 are shown the respective transfer curves at pH 6 for Dispersol Fast Scarlet B, and it is seen that acetylation has practically no effect on the uptake of the disperse dye. In Fig. 11 is a comparison of the transfer of Solacet Fast Scarlet B and each of the three acid dyes on normal and acetylated nylon respectively. This should be studied in conjunction with Fig. 12, which relates the equilibrium adsorption of each of the dyes with the free amino-group content of the nylon, including results with the ICI nylon sheet (0·033 g.-equiv. NH_2 per kg.). Adsorption is thereby shown to be directly proportional to the number of amino groups available. If each of the curves is extrapolated to zero amino-group content, there is shown to be a finite exhaustion for each of the dyes, and this is the

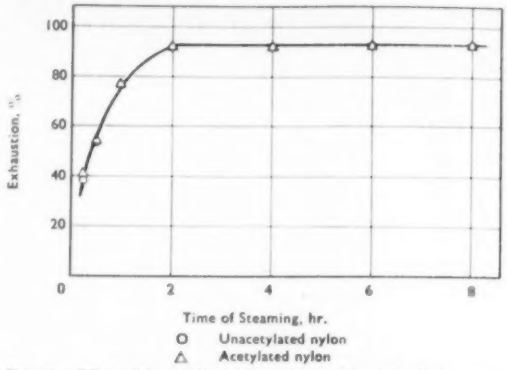


FIG. 10—Effect of Acetylation of Free Amino Groups in Nylon upon Transfer of Pure Dispersol Fast Scarlet B from Starch to Nylon on Steaming

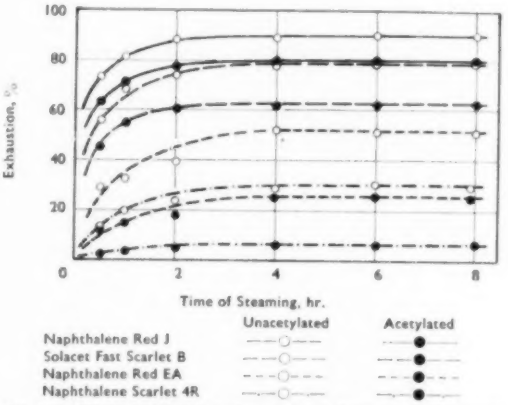


FIG. 11—Effect of Acetylation of Free Amino Groups in Nylon upon Transfer of Solacet and Acid Dyes from Starch to Nylon during Steaming

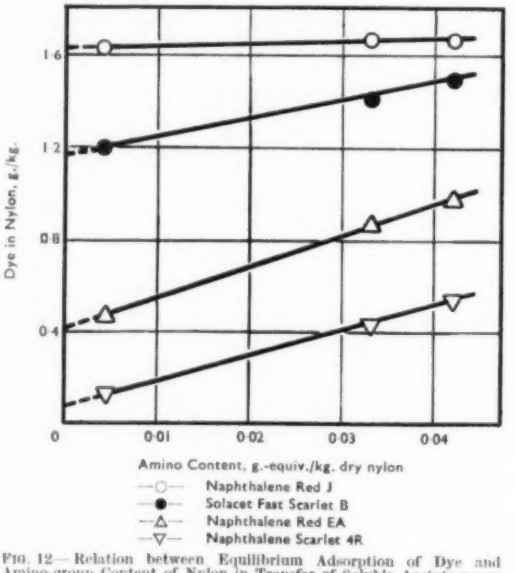

Dye in Nylon, g./kg.
Amino Content, g.-equiv./kg. dry nylon
—○— Naphthalene Red J
—●— Solacet Fast Scarlet B
—△— Naphthalene Red EA
—▽— Naphthalene Scarlet 4R

FIG. 12—Relation between Equilibrium Adsorption of Dye and Amino-group Content of Nylon in Transfer of Soluble Acetate-rayon and Acid Dyes from Starch to Nylon during Steaming

measure of that part of the adsorption for which chemical forces other than electrovalencies are available. With Naphthalene Red J this proportion is high, and the fraction of the exhaustion on the unacetylated du Pont nylon for which electrovalencies could be responsible is shown to be about 3% only.

In Table I are collected the results of transfer experiments (at pH 4) in which equilibrium conditions were set up with each of the three acid dyes when the initial dye content of the starch film exceeded that required to neutralise all the free amino groups in the nylon.

TABLE I

Dye Transfer to Nylon

(Dye transferred, g.-equiv. per kg. nylon)

Nylon, g.-equiv. NH_2/kg. ...	...	0·033	0·042
Naphthalene Red J ...	...	0·048	0·050
Naphthalene Red EA ...	...	0·035	0·042
Naphthalene Scarlet 4R ...	...	0·025	0·036

The agreement shown between the equivalents of NH_2 in the fibre-substance and the equivalents of dye taken up is very striking for Naphthalene Red EA, which is the dye of medium water-solubility, but this agreement must be considered fortuitous to some extent in view of the evidence that electrovalencies are not wholly responsible for the adsorption of the dye. Nevertheless, there is a satisfying explanation for the greater departure from equivalence of the two other acid dyes, the one because its solubility in water is relatively high, the other because the solubility is relatively low.

EFFECT OF ADDED ELECTROLYTE

The effect of adding of sodium chloride to the printing paste from which the starch films are made is shown in Fig. 13 for Solacet Fast Scarlet B, without the addition of buffering agents, and in Fig. 14 for Naphthalene Red EA at pH values of 2·2 and 6·8 respectively. The results call for little comment, and it will be seen that, as expected, for Naphthalene Red EA the effect of neutral electrolyte is greatest at the lower pH, where attachment of dye molecules to the nylon by electrovalencies is most in evidence.

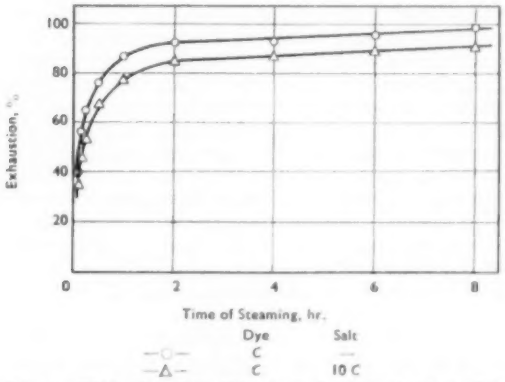


FIG. 13—Effect of Sodium Chloride on Transfer of Solacet Fast Scarlet B from Starch to Nylon on Steaming ($C = 0·64$)

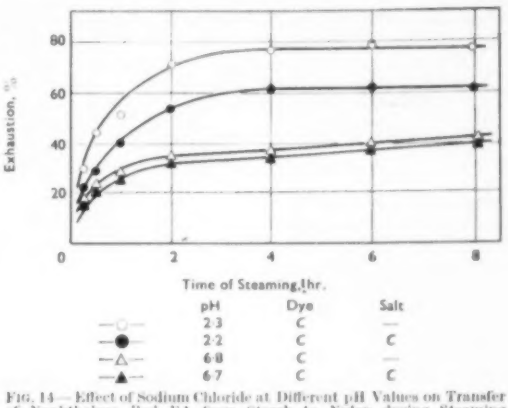


FIG. 14—Effect of Sodium Chloride at Different pH Values on Transfer of Naphthalene Red EA from Starch to Nylon during Steaming ($C = 0·63$)

DESORPTION OF ACID DYE FROM NYLON TO STARCH

In these experiments, strips of nylon film were combined with a starch film, containing Naphthalene Red EA, and buffered to pH 2·2. Steaming was continued until equilibrium transfer of dye to the nylon had been reached. The two kinds of film were then separated, and the nylon film, containing migrated dye, was combined with a starch film containing sodium chloride but no dye (pH of the starch paste used in preparation: 6·8), and again steamed.

TABLE II

Desorption of Dye from Nylon

Time of Steaming (hr.)	Desorption (%)
1	16
2	26
3	39
4	41
6	42
8	42

It is evident from Table II that desorption of dye from the nylon can be appreciable. The proportion of dye remaining in the nylon film at equilibrium is slightly less than that required to neutralise all the free amino groups present.

KINETICS OF DYE TRANSFER

The curves representing the course of dye transfer from starch to nylon (Fig. 1, 2, 4) are approximately hyperbolic in form, and by a suitable choice of constants can be represented by the equation—

$$kt = \frac{1}{A - x} - \frac{1}{A}$$

(k = velocity coefficient; t = time of steaming; A = equilibrium exhaustion in which the dye transferred to the nylon is expressed as a percentage of total dye present; and x = the corresponding percentage exhaustion at end of time t). In Table III are set out the values for the velocity coefficient which give the closest agreement between calculated and experimentally determined rate-of-transfer curves for both disperse and soluble

dyes for acetate rayon in those experiments where equilibrium could be established.

TABLE III

Velocity Coefficients of Dye Transfer

Dye	Initial Wt. of Dye in Starch (mg./unit disc)	Velocity Coefficient ($\times 10^{-5}$)
Dispersol Fast Scarlet B ...	0·155	55·8
	0·102	72·2
	0·028	196·0
Solacet Fast Scarlet B ...	0·163	96·7
	0·089	129·0
	0·041	360·0
Disperse Dye A	0·099	12·0
Soluble Dye A	0·250	99·0
	0·076	260·0

Thus (*a*) for all dyes except Disperse Dye A, where information is not complete, the velocity constant increases with diminishing initial concentration of dye in the starch phase; (*b*) in the pair of homologous disperse dyes, the rate of transfer of the one with lower molecular weight is specifically the greater; (*c*) in the corresponding pair of soluble dyes, the one of higher molecular weight is transferred the more rapidly; and (*d*) at comparable initial concentrations in the starch, the rate of transfer of a disperse dye is less than that of the corresponding soluble dye.

Discussion

As a measure of dye transfer, through a given area of contact, between a thickener phase of relatively small volume and a nylon phase of relatively large volume, the present results have no exact counterpart in dyeing investigations. Nevertheless, in spite of the differences in working conditions, they follow, in all essential features, the corresponding results in dyeing investigations for the examples of disperse, soluble acetate-rayon, and acid dyes which have been examined. Nowhere, in fact, have results been obtained which could not have been predicted from precedent knowledge of the dyeing behaviour of nylon. Thus, although one or two topics call for extended discussion, it is first convenient to compare briefly the present results with those already obtained by other workers in the dyeing field.

DISPERSE DYES

EXHAUSTION—In dyeing experiments, exhaustion can vary from 50 to 100% for a variety of commercial brands [2], and the value for Dispersol Fast Scarlet B reaches 92%. In the present work, maximum observed exhaustion of the same dye after purification is 97%, and that of the higher homologue, Disperse Dye A, 88%. In both types of system, equilibrium exhaustion values are independent of the amount of dye in the system up to the point at which saturation of the nylon occurs, so that the "solid solution" dyeing mechanism, which is indicated for printing systems with cellulose acetate as the fibre-substance [1], is indicated for nylon also.

RATE OF TRANSFER—Although typical dyeing systems differ from the present experimental printing system in (*a*) relative dimensions of the two phases, (*b*) presence or absence of starch in the non-fibre phase, (*c*) molecular arrangement in the fibre-substance phase, (*d*) total proportion of water present, the rates of dye transfer are surprisingly similar. For example, with Dispersol Fast Scarlet B, the range of velocity constants is 50–730 $\times$ 10^{-5} for dyeing and 55–200 $\times$ 10^{-5} for printing. Among the homologous dyes examined, the velocity constant in both systems diminishes with increasing molecular weight [2].

IONISATION OF TERMINAL AMINO GROUPS—In both dyeing and printing, the rate and the extent of dye transfer are little affected by changes in the pH of the medium [14], or by the blocking of terminal amino groups by acetylation [15]. The conclusion arrived at in studying dyeing processes, that the principal attraction between dye molecules and fibre-substance is not that which exists between charged ionic centres, is confirmed for printing transfers also.

Because of the absence of strongly ionised groups in the dye molecule, and because the proportion of the disperse dye which can be in solution at any one time is small, the influence of the surface potential of the fibre-substance and that of the osmotic pressure of mobile gegenions upon the course of dye migration are likely to be very small. Thus, although the dye is presented to the fibre-substance at much higher concentrations in the printing system than in normal dyeing systems, the rate and the extent of transfer are little different. In this respect, transfer of the disperse dyes to nylon departs greatly from, say, the transfer of direct dyes to cellulose [6].

SOLUBLE DYES FOR ACETATE RAYON

With this class, also, the transfer behaviour of the dyes is very similar in dyeing and in printing systems respectively. When the behaviour of acid dyes has been discussed, it will be seen that the soluble dyes for acetate rayon share some of the properties of the disperse dyes and some of those of the acid dyes.

EXHAUSTION—In dyeing experiments, the maximum attainable exhaustion with a variety of commercial brands has been given as 75–100% [16], though in some cases these values are reached only after additions of acetic acid to the dyebath [17]. In the present work, exhaustion of the pure dye from a neutral starch film reaches 98% for Solacet Fast Scarlet B and 96% for the higher homologue Soluble Dye A. The higher values with the pure dyes in the printing experiments may be ascribed in part to the absence of electrolytes, which depress exhaustion, and in part to the high concentration at which the dye is presented to the nylon.

EFFECT OF pH—In sharp distinction from the behaviour of systems in which cellulose acetate is the fibre-substance, both the rate and the extent of dye transfer to nylon are greatly affected by the pH. Depression of pH into the region in which the majority of the terminal amino groups become ionised enhances dye transfer both in dyeing and in printing. Correspondingly, acetylation of the terminal amino groups causes a proportionate suppression of the adsorption even from acid

media. Finally, the presence of neutral electrolyte, through the provision of anions which compete with the dye ions for the cationic centres in the nylon, depresses adsorption, particularly at low pH values.

ACID DYES

As in dyeing, the rate and the extent of dye transfer to the nylon are shown to be strongly dependent upon the pH of the system, the effect of a given change in pH being different for different dyes [3,4,18,19]. Peters [3] has given a curve for the equilibrium adsorption by nylon of selected acid dyes from aqueous baths at different pH values. This should be compared with Fig. 9. Maximum adsorption values between pH 3 and 4 seem to correspond to the saturation of the ionised primary amine groups in the nylon. In printing as in dyeing, the specific effect of the pH of the system varies from dye to dye. Where the proportion of $-SO_3Na$ groups in the dye molecule is low, e.g. Naphthalene Red J, the rise in adsorption as the pH is diminished is less than when this proportion is high (e.g. Naphthalene Scarlet 4R) and adsorption is appreciable even at pH 10 (Fig. 8). The rate of dye transfer varies in the same order. Thus, as with the soluble dyes for acetate rayon, adsorption of acid dyes by nylon is again shown to be the resultant of at least two different mechanisms. One is the process which depends upon the attachment of dye molecules through electrovalencies to ionic centres in the fibre substance, $-NH_3^+$ in the region of pH 3–4, and also $>NH_2^+$ at lower pH values. This mechanism comes increasingly into play as the pH is lowered and as the number of $-SO_3Na$ groups in the dye molecule increases. The other mechanism is similar to that by which disperse dyes are attached to the nylon, and has been ascribed to the formation of hydrogen bonds between active groups in the dye molecule and the peptide (CO·NH–) groups in the nylon [20]. This mechanism is preferentially in operation at high pH values, with dyes which have a low proportion of $-SO_3Na$ groups in the molecule, and with nylon in which the terminal amino groups have been acetylated. It is little influenced by the presence of neutral electrolyte.

There has been much discussion about the mechanism of the dyeing of nylon with acid dyes, and the original papers should be consulted [3,4,19,21–23]. One of the most recent disputes has been that of Remington and Gladding [23] with workers in this country [3,21,22,24,29] about the precise significance of the large rise in adsorption as the dyebath becomes strongly acid. The latter ascribe it primarily to increasing ionisation of the weakly basic secondary amino (>NH) groups in the polyamide chain of the nylon [3,22] Remington and Gladding [23] to progressive hydrolysis of –CO·NH– groups in the same chain, setting free more and more primary amino groups. In all this work there has been a conflict of interpretation rather than of observed results. The present work with the printing system, because it has produced nothing which seriously contradict the results of investigations of dyeing processes, has added very little, at this stage, to knowledge of the mechanisms by which acid dyes are taken up by nylon. It has, however, with the acid dyes selected, shown no evidence to suggest an essential difference between the manner of dye transfer to the fibre-substance during steaming of a print and the manner of transfer to the fibre in a conventional dyeing operation.

PARTITION OF DISPERSE DYES

The arguments for and against the theory of solid solution are very similar to those which were discussed in the preceding paper dealing with the corresponding system for cellulose acetate [1]. The argument again rests partly on analogy, the evidence of constant partition of a number of organic solutes between nylon and water [25,26], and partly on the evidence of a widespread independence of initial dye concentration in the exhaustion of the disperse dyes at equilibrium [2,25]. The main doubt about the existence of a solid solution mechanism has been expressed by Vickerstaff and Waters [27], and the arguments are the same as those which have been applied to dyeing systems where cellulose acetate is the fibre. In the present work also, the experimental evidence, in so far as it shows the existence of a constant dye partition between starch and nylon at equilibrium over a wide range of initial dye concentrations, and a sharp inflexion of the isotherm corresponding to the point of saturation, supports most the idea of solid solution. The arguments by which Vickerstaff's objections are shown to require qualification are the same as those adduced for the cellulose acetate printing system, since they are directed more to the state of the dye in the starch phase than to its state in the fibre phase [28].

* * * *

Our best thanks are due to Messrs. Imperial Chemical Industries Ltd., Dyestuffs Division, of Manchester 9, for valuable help in the provision of dyes, intermediates, and nylon in sheet form; to Messrs. E. I. du Pont de Nemours & Co. of Buffalo, N.Y., also for the provision of nylon sheet; to the Foreign Education Committee of Bombay and to the Manchester Education Committee for grants which have enabled one of us (E.H.D.) to take part in this work.

DEPARTMENT OF TEXTILE CHEMISTRY
COLLEGE OF TECHNOLOGY
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Notes

Proceedings of the Council

At a meeting of the Council held at the Offices of the Society, 19 Piccadilly, Bradford, on 9th September 1953, the proceedings included the following items of interest—

EASTBOURNE COLLEGE SCIENTIFIC SOCIETY— It was reported that Dr. H. W. Ellis had kindly agreed to give to this Society a lecture on dyes and dyeing.

DYERS' CO. MEDAL COMMITTEE— It was reported that the Worshipful Company of Dyers had nominated Dr. C. J. T. Cronshaw to represent the Company on the Research Medal Adjudication Committee.

MEMBERSHIP— Six applications for ordinary membership and one for junior membership were approved.

Meetings of Council and Committees October

Council— 21st
Finance— 21st
Publications— 20th
Fastness Tests Co-ordinating— 1st and 29th
Colour Index Editorial Panel— 6th
Terms and Definitions— 2nd
Society's Medals— 5th
Review of Textile Progress— 26th
Joint Conference— 29th

John Barritt, B.Sc., A.R.C.S., A.R.I.C. Honorary Member

At the meeting of Council held on 21st October 1953, it was unanimously resolved that Mr. J. Barritt be made an Honorary Member of the Society, in recognition of his great services to the Society as Honorary Secretary from 1946 to 1953.

Deaths

We regret to report the loss by death of Messrs. J. V. Armstrong and E. L. Duhring.

Tentative Definitions

The Terms and Definitions Committee will welcome comments on the following tentative definitions*—

35. ACID DYE

An anionic dye characterised by its substantivity for protein fibres, and normally applied from an acid dyebath.

36. ANIONIC DYE

A dye which dissociates in aqueous solution to give negatively charged coloured ions.

37. BACKWASHING

The washing of dyed or undyed wool sliver before or after gilling or combing.

38. BASIC DYE

A cationic dye characterised by its substantivity for tannin-mordanted cotton (see also Cationic Dye).

39. CATIONIC DYE

A dye which dissociates in aqueous solution to give positively charged coloured ions (see also Basic Dye).

40. COLLOIDAL DYES*

Dyes which in aqueous solution under specified conditions exist mainly as aggregates, only a small proportion being present as single molecules or ions.

41. DIFFUSION

A movement of molecules or ions through a solution or fibre due to the existence of a concentration gradient.

42. DIRECT COTTON DYE (DIRECT DYE)

An anionic dye having affinity for cellulosic fibres when applied from an aqueous dyebath containing an electrolyte.

43. DYE-FIXING AGENT

A product which is capable of reacting with a dye on a fibre to give improved fastness to water or to washing.

Note— Dye-fixing agents are normally applied as aftertreatments to dyes which already have some affinity for the textile substrate and are so distinguished from mordants.

44. JIG (JIGGER)

A dyeing machine in which fabric in open width is transferred repeatedly from one roller to another through a dyebath of relatively small volume.

45. LIQUOR RATIO

The ratio of the weight of liquor employed in any treatment to the weight of material treated.

46. MATCHING

The process by which the proportion of each dye present in a material is adjusted so that the final colour resembles that of a given sample as closely as possible.

47. MÉLANGE PRINTING

A printing process in which bands of thickened dye paste are applied, with intervening uncoloured areas, across wool or other slubbing. The slubbing is subsequently steamed, washed, and then combed to produce an even mixture of dyed and undyed lengths of fibre.

48. TIPPY WOOL

Wool in which the tip portions of the fibres have been so damaged by natural weathering as to have markedly different dyeing properties from the root portions.

* The above definitions have been considered by the Textile Terms and Definitions Committee of the Textile Institute, which is in agreement except in the case of No. 40.

Worshipful Company of Dyers

Mr. Cyril R. Egerton has been elected Prime Warden, and Mr. R. P. Chambers, F.C.A., has been elected Renter Warden.

Sales of Household Dyes in the U.S.A.

The value of the sales of household dyes in the U.S.A. has increased from $8,000,000 in 1949 to $12,600,000 in 1952. COC

Scottish Section
Annual Report 1952–1953

We are pleased to report another successful session, during the course of which nine meetings were held— eight in Glasgow and one in Galashiels. With the exception of one meeting in Glasgow, attendances have been exceptionally good.

Although we enrolled ten new members during the session, our total shows only an increase of two as compared with the previous year, this being due to resignations and transfers to other Sections.

At the Annual General Meeting of the Section Messrs. T. Brown, C. H. Giles, J. G. B. McCallum, and P. Schwarzenbach retired from Committee by rotā. Mr. Brown had intimated that he did not wish to be nominated for re-election and, accordingly, Mr. H. R. Crone was nominated to fill the vacancy. This gentleman, together with Messrs. Giles, McCallum, and Schwarzenbach, was duly elected.

Arrangements are well in hand for the 1953–54 programme, and during the second half of the session it is hoped to arrange an interesting meeting to which ladies will be invited.

F. ATACK
Honorary Secretary
Scottish Section

Society of Dyers and Colourists of Australia
Annual Report 1952–1953

The Council has been pleased to note the manifestation of increased activity in the Society during the year ending 31st May 1953.

The Council met twice under the chairmanship of the President, Mr. W. C. Castle. Several decisions were reached which should prove beneficial to the Society.

Mr. J. A. Eastwood, the Immediate Past President, was presented with a replica gavel as a token of esteem for the long and valuable service rendered to the Society. Mr. J. A. Eastwood founded the New South Wales Section of the Society.

The Council occupied itself with the history of the Society, and steps are being taken to have this work brought up to date.

A new Section of the Society was founded in Queensland on 12th September 1952. Earlier, Mr. J. A. Eastwood had written to Mr. J. R. Hinchley in Brisbane suggesting that there would probably be sufficient persons in Queensland interested to become country members attached to the N.S.W. Section. Through the activities of Mr. Hinchley and Mr. C. Keyvar, the number of these country members increased until it was decided to form a Queensland Section. A meeting was arranged after approval had been granted by the Council. This meeting was attended by the President, Mr. W. C. Castle, whose assistance was of immense value in launching this new venture.

The Society has now 347 members, but it is hoped that membership will increase, as the number of new members being enrolled is, so far, not making up for the losses of members brought about for various reasons.

All Sections report successful evenings throughout the year. A very high standard was maintained in the lectures, "quiz" sessions, and the film evenings, which were all much enjoyed.

Several happy social functions were organised by the Sections during the year. Annual Dinners were held by the Victorian, New South Wales, and South Australian Sections. The N.S.W. held their annual ball and the Victorian Section held a country week-end at Hepburn Springs.

Gratitude is expressed for the efforts of Mr. L. P. Rendell, chairman of the publication committee, in arranging the publication of lectures and the activities of the Society.

The Society is indebted to the authors of lectures who have permitted these to be published, and to Mrs. A. Mardell, Editress of the *Textile Journal of Australia*, for her kind co-operation.

The President, Mr. W. C. Castle, visited all Sections during the year and has been impressed with the work they are doing in the interests of the Society.

B. FIRTH
Honorary General Secretary

OFFICERS AND COMMITTEES 1953–54

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Immediate Past President ...	J. A. Eastwood
Honorary General Treasurer ...	J. A. Fergusson
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Manufacturers' Publications and Pattern Cards

The Society does not accept any responsibility with regard to the statements in the following notes. Any publication abstracted may be referred to by members of the Society on application to Dr. C. B. Stevens, Dyeing Department, Leeds University

Badische Anilin- und Soda-Fabrik

VIALON FAST DYESTUFFS ON PERLON SLUBBING—This card contains dyeings in three depths on Perlon (nylon) slubbing of a range of sixteen Vialon Fast dyes together with thirty mixture dyeings. This class of dye is applied in the normal manner, dyeing being commenced at a low temperature in the presence of ammonia and Uniperol PN. The dyeliquor is raised to boiling point in 30 min. and dyeing continued at the boil for 2–3 hr. during which time ammonium sulphate is added to promote exhaustion. Dyeings are characterised by good fastness to light and particularly to wet treatments and the range is recommended for dyeing superpolyamide fibres in slubbing form or as loose fibre to be subsequently converted into yarn and cloth alone or after mixing with other fibres.

Cassella Farbwerke Mainkur

VAT DYESTUFFS IN MACHINE PRINTING—This card contains prints in two depths on mercerised cotton and viscose rayon crêpe of thirty-four vat dyes including twenty Indanthrens, five Algols, two Hydron Blues, two Immedials, and three Indocarbon blacks.

SOLIDOGEN FFL—This auxiliary product is designed to increase the wet fastness properties of dyeings of direct, sulphur, Union, Union Fast, and Union Metachrome dyes. No significant change in either the hue or the fastness to light of the dyeings occurs and a particular advantage is that the treatment is carried out at room temperature using a liquid product which is miscible with water in any proportion. The card contains straight and aftertreated dyeings of direct dyes, aftertreated direct dyes (including aftercoppering, formaldehyde and diazotisation and development), Immedial and Union dyes together with patterns of both after various washing, water, soda boiling and wet-ironing tests. A range of discharge prints on viscose rayon crêpe illustrates the value of Solidogen FFL for improving the fastness to water of this type of material.

The Clayton Aniline Co. Ltd.

DYES FOR UNION MATERIALS—This card contains dyeings on wool–cotton mixture cloth of 182 dyes classified as follows: one hundred and twenty-one direct dyes (including forty-four Chlorantine Fast and twenty-three Coprantine dyes) applied at (*a*) 60°C. in the presence of Albatex WS to give the maximum reservation of the wool portion compatible with covering the cellulosic fibre and (*b*) at 100°C.; twenty-three Rosanthrene and Diazo dyes diazotised and developed on the fibre with Yellow Developer C, *β*-naphthol and *m*-toluylene diamine where applicable; and thirty-seven neutral dyeing acid dyes.

DYESTUFFS FOR NYLON—This card contains dyeings on nylon cloth of ninety-three dyes including the following: eighteen disperse dyes from the Cibacet range; twenty-six acid dyes including eleven Cloth Fast dyes; seven direct dyes applied from an ammonium sulphate dyebath; eighteen Neonyl dyes—these are a group of premetallised dyes applied in the presence of 3–4% of formic acid or sulphuric acid, the latter producing more level results in pale shades; fourteen chrome dyes applied in the presence of formic acid and chromed at the boil for 1½–2 hr. using an amount of dichromate equal to the weight of dye up to the maximum of 2% and 1–3% of sulphuric acid. In the case of the water soluble dyes, the data supplied include a figure for the percentage of dye taken up by the fibre at saturation point. The card also contains thirty dyeings of mixtures of dyes from the disperse, acid and Neonyl ranges.

NEOLAN DYES: STANDARD AND FASHION SHADES ON WOOL PIECE—This card contains dyeings in three depths of a range of thirty-six premetallised dyes of the Neolan type and forty-two dyeings of mixtures of dyes from this group.

DIRECT AND CHLORANTINE FAST DYESTUFFS ON COTTON PIECE—This card contains one hundred and seventy-four pairs of dyeings of direct dyes including sixty Chlorantine Fast Dyes. In six instances, dyes giving dyeings of poor fastness to storage are represented by matchings produced using faster dyes. Data appended alongside each set of dyeings includes the S.D.C. dyeing classification.

CIBANTINE GREY 3G—This solubilised vat dye gives greys of excellent fastness on cellulosic fibres. It is recommended for use alone and in combination with Cibantine dyes in affinity groups I and II, an example of such use being the production of silver grey in conjunction with Cibantine Blue GF. Fastness figures on cotton include—Light 6–7, soda boiling 4–5, chlorine 5.

COPRANTINE YELLOW RL—This leaflet is Supplement No. 1 to the card "Coprantine Dyestuffs in Screen Printing". Yellow RL is particularly recommended for heavy prints on cellulosic fibres, either as a self colour or in mixtures, where good fastness to washing is required. Fastness figures for prints on cotton include—Light 5, washing 5.

CIBALAN DYEINGS ON SLUBBING AND YARN—This card contains dyeings in two depths on slubbing and yarn of seventeen Cibalan dyes. The range includes two yellows, an orange, a scarlet, a red, three bordeaux, a violet, two blues, three browns, a khaki and two greys. Twenty-seven mixture dyeings on slubbing and an equal number on yarn are also included.

ALIZARINE FAST GREY BLL—This fast-to-milling acid dye gives greenish greys of good fastness to light and wet treatments and is particularly recommended for greys on loose wool and slubbing. Only slight dulling occurs in the presence of dichromate and it is thus of interest for shading chrome dyes when using either the on-chrome, afterchrome, or single-bath methods. Fastness grades include—Light 6–7, water milling 5, perspiration 3.

CHLORANTINE FAST BLUE 2BLL—This direct dye is particularly recommended for dyeing yarn, knitted and woven cloth and furnishing fabrics, particularly materials which are to be given a crease-resist finish. "Barry" viscose is covered well; acetate rayon is reserved. It is also suitable for direct printing but dyeings are not dischargeable. The wet fastness of dyeings is somewhat improved by an aftertreatment with Lyofix SB conc. and considerably so when Coprantex A is used. The application of either product produces only a slight reduction in light fastness but the hue becomes slightly greener and duller with Coprantex A. Fastness figures on cotton include—Light 6, washing (at 40°C.) 3–4, perspiration 4–5.

COPRANTINE YELLOW RL—This direct dye gives bright yellows of good fastness on cellulosic fibres. Dyeings are unaffected by crease-resist finishing and dischargeable to white. Dyeings are markedly redder and faster to light than those using Coprantine Yellow 2G. Fastness figures on cotton include—Light 5, perspiration 4, washing (at 50°C., soap + soda) 5.

CIBANTINE YELLOW 2GB—This solubilised vat dye gives full bright yellows of excellent fastness to washing and hypochlorite and good fastness to light when printed on all types of cellulosic material. It is suitable for direct and resist printing and also for bright yellow resists under aniline black and Naphthol Blue; dyeings are also dischargeable to a pure white using a Formosul—anthraquinone discharge. Fastness figures on cotton include—Light 4–5, chlorine (wet) 4–5, perspiration 4–5.

CIBANTINE ORANGE 8R—This solubilised vat dye gives reddish oranges of good fastness to light and wet treatments on all forms of cellulosic material. It is recommended for application on the pad or jig, alone and in mixtures, particularly with Cibantine Brown BR or 3B for pinks, browns and beiges. Fastness figures on viscose rayon include—Light 7, washing (at 100°C.) 4–5, chlorine 5.

Durand & Huguenin A.G.

INDIGOSOL YELLOW 2GB—This solubilised vat dye is recommended for printing to give yellows, the hue of which lies between that given by Indigosol Golden Yellow IGK and Yellow V. It may be printed on naphtholated cloth, without steaming, alongside diazotised bases by

using the chromate/oxalate method of development. Dyeings can be discharged to a perfect white using a Hydrosulphite NF conc.— Leucotrope W discharge and it is easily reserved, Reserve X being unnecessary. Fastness figures on cotton include— Light 4–5, chlorine 5, washing (3 hr. at 100°C.) 4–5.

Farbenfabriken Bayer

EULAN AWA— This pamphlet describes the properties and uses of a concentrated mothproofing agent particularly designed for applying from a cold or lukewarm solution to all types of keratinous materials by simple impregnation. It may be applied during washing or milling, by padding or dipping, and the mothproof finish is fast to water, perspiration, light, and weather. It has marked bactericidal properties and treatment also confers considerable protection against attack by mildew and mites.

The Geigy Co. Ltd.

DYESTUFFS FOR SLUBBING AND LOOSE WOOL— This is a particularly elaborate pattern card containing dyeings on slubbing of 109 dyes including 68 chrome, 26 Polar, 4 Erio Fast, 3 Brilliant Wool and 8 Irgalan dyes, each (with the exception of blacks) being represented in five depths. Relevant fastness data are appended alongside the dyeings and the text includes details of the fastness tests employed and lists of dyes recommended for use where fastness to peroxide bleaching, to formaldehyde in the dyebath, to sulphite, and to acid planking, is required.

DYESTUFFS FOR PAPER— This card contains dyeings in two depths of one hundred and twenty-two dyes most commonly used for dyeing paper in the beater. They include thirty-five direct dyes, twenty-five Solophenyl and Paper Fast dyes, thirty-three acid dyes, seventeen basics and twelve pigments including six Tinofil colours and six Tinon and Tinon Chlorine (vat) dyes which may be considered as pigment colours when applied to paper by direct incorporation.

IRGALAN DYESTUFFS— This card contains dyeings in five depths on wool cloth of eleven Irgalan (premetallised) dyes including two yellows, an orange, a red, a bordeaux, three browns, an olive and two greys, together with three Irganol S dyes. Unlike the Irgalans, the latter are not metal-dye complexes but their similarity in dyeing properties and the fastness and brightness of the dyeings obtained make them useful as supplementary and shading colours. Dyeings on nylon slubbing of the Irgalan range only are also included. The card contains a comprehensive selection of mixture dyeings including, on wool, thirty-two mixtures on slubbing, forty-eight on yarn, twenty-four on gabardine cloth and twenty-eight on ladies' dress material together with eighteen mixtures on nylon slubbing.

L. B. Holliday & Co. Ltd.

SEMI-CHROME SHEEPSKIN SUÈDE— This card contains dyeings of 60 acid dyes. Information appended alongside each set of patterns includes fastness to light, acid and alkali; solubility at 50°C. and an assessment of the levelling power, penetration and bleeding in fat liquor.

BLANCOL BT— This is an optical bleaching agent designed for use in detergent preparations. It possesses good affinity for cellulosic fibres when applied under neutral or alkaline conditions but is not applicable to acetate rayon, nylon or wool.

Imperial Chemical Industries Ltd.

ACRONOL PHLOXINE FF— This basic dye gives bluish reds of exceptional brilliance on tannin-mordanted cellulosic materials and is of interest when brightness is of greater importance than other fastness demands. It may also be applied to material mordanted with Taninol BMN, although in this case the colour is duller. It has good solubility and may be applied to all forms of yarn and cloth. It may be applied to silk and is useful for bright shades on jute, coir and sisal and is also of some interest for textile printing. Fastness figures on tannin-mordanted cotton include—Light 1–2, washing (mild, once at 40°C.) 3–4, water 3.

BRENTHOL BC— This new coupling component for azoic dyeings gives a range of colours on cellulosic materials ranging from bright oranges to scarlets and bordeaux. Principal advantages claimed include good solubility when dissolved by either the hot or cold methods; high substantivity; excellent stability of the impregnated material to atmospheric exposure even in the absence of formaldehyde; complete solution can be maintained using a relatively low concentration of caustic soda; dyeings having good fastness properties can be obtained in combination with a wide range of diazo components. The text includes graphical representations of its substantivity at 25°C. in the absence and presence of salt when applied to unmercerised cotton, mercerised cotton, and viscose rayon, respectively. Fastness grades of a standard depth dyeing on cotton using Brentamine Fast Yellow GC include— Light 5, soda boiling 4, hypochlorite 5.

LISSAMINE ULTRA BLUE AR— This equalising acid dye gives bright blues of good fastness to light on wool. In level dyeing properties it is superior to the majority of anthraquinone acid blues while dyeings are faster to sea water and "salt spotting" than those obtained using the level dyeing anthraquinone blues. It is of particular interest as the blue component in mixtures on yarn and knitted and woven cloth and also on carbonised material. It is suitable but only of limited interest for direct printing on wool, silk and nylon. It is of no interest for printing viscose rayon by the urea process and is not suitable for discharge printing. Fastness figures on wool include— Light 5–6, washing (mild once at 40°C.) 3, perspiration (pattern at pH 7·8) 3.

POLYMON YELLOW RS— This is a new type of pigment designed and controlled for colouring plastic moulding materials. It is very stable to heat and readily dispersible from the dry powder state and gives colourings having good fastness to light. At moderate concentrations, transparent effects are obtained which are visually indistinguishable from those using soluble dyes. Fastness figures for a coloured polystyrene plastic using 0·1% of dye include— Light 7, heat stability (280°C. for 15 min.) little weaker.

MONOLITE FAST BLUE 3 RS— This is a reddish blue pigment of high tinctorial strength and excellent fastness to light, heat, acids, alkalis, and lime. It is redder in hue than Monastral Fast Blues BS and LBS but appreciably greener and duller than Fastel Blue B. It is recommended for colouring a very wide range of materials including paints, lacquers, stoving enamels, soap wrapper and carton inks and nitrocellulose leather cloth and leather finishes. Fastness to light tested as a full shade in linseed oil medium, on a 1–10 scale (madder taken as 10), 10.

Sandoz Products Ltd.

RESOFIX COLOURS ON COTTON— This card contains dyeings in two depths on cotton cloth of twenty-two Resofix dyes. In all cases the dyeings have been after-treated with Resofix CU to develop the maximum fastness to wet treatments. The dyes themselves have been divided into two main groups. The first group are superior in migrating power to the second group and may be dyed in short liquors in circulating liquor machines; the second group, which are denoted by the suffix "C", exhibit higher fastness but in some cases have poorer migrating properties. Mixtures containing dyes from both groups are not recommended. The relevant fastness data are given alongside each dye together with a graphical representation of the dyebath exhaustion in both the presence and absence of salt.

BRILLIANT ALIZARINE MILLING RED FBL— This acid dye giving bright bluish reds of good fastness to light and wet treatments is recommended alone and in mixtures for dyeing wool in all forms and is also of interest on nylon and weighted and unweighted silk. Together with Brilliant Alizarine Milling Violet FBL and Blue FGL which are similar in fastness properties and brightness, it is suitable for shading Lanasyn dyes and brightening chrome dyeings. Fastness figures on wool include— Light 6, washing (60°C.) 4, alkaline milling 4.

BRILLIANT ALIZARINE MILLING VIOLET FBL— This acid dye gives exceptionally bright violets on wool, silk and nylon, having good fastness to light and wet treatments. Recommended uses and dyeing characteristics are the same as for Brilliant Alizarine Milling Red FBL described above. Fastness figures on wool include— Light 6, washing (60°C.) 4, alkaline milling 4.

TANASYN YELLOW 2RL—This metalliferous dye is an addition to the range of neutral dyeing dyes recommended for dyeing loose wool, yarn and cloth and mixtures of wool and superpolyamide fibres as loose stock, slubbing and yarn where high fastness to light and wet treatments is demanded. Used alone it gives brownish-yellows while in mixtures with the corresponding Brown RL, Grey BL, Orange RLN and Green BL, beiges, browns, greys and greens having the same excellent fastness properties can be produced. It is also of interest for direct printing on wool, silk and nylon. Fastness figures on wool include—Light 7, washing (60°C.) 4–5, alkaline milling 4–5.

SOLAR GREEN 3LB; LUMICREASE GREEN 3LB—This direct dye gives bluish greens of good fastness to light on cellulosic fibres and is particularly recommended for use on curtain and finishing fabrics alone and in combination with selected members of the Solar range. The fastness of dyeings to wet treatments is considerably improved by an aftertreatment with Sandofix WE, the hue becoming somewhat bluer. As indicated by the alternative name, crease-resist finishing does not impair the fastness to light of the dyeings. Fastness figures on cotton include—Light 7, washing (medium depth dyeing, 40°C.) 2–3, perspiration 2–3.

SOLAR GREY 3LB; LUMICREASE GREY 3LB; CUPROFIX GREY 3LB; RESOFIX GREY 3LB—The versatility of this direct dye is indicated by the alternative names under which it is marketed. Bluish greys of good fastness to light are obtained on direct dyeing. Aftertreatment with Cuprofix S new or Resofix CU considerably increases the fastness of dyeings to wet treatments, Resofix CU producing the greatest improvement. The fastness of dyeings to light is unaffected by normal crease-resist finishing. Fastness figures for a dyeing on cotton aftertreated with Resofix CU include—Light 6–7, washing (medium depth dyeing, 100°C.) 4, perspiration 3.

XYLENE FAST ORANGE P—This addition to the "P" series of acid dyes for wool gives bright oranges of good fastness to wet treatments. It is particularly recommended as a component for browns on hosiery. Solid dyeings are readily obtainable on wool/nylon mixtures. Cellulosic and acetate rayon effect threads are reserved white. Fastness figures on wool include—Light 5, washing (40°C.) 4–5, perspiration 3–4.

FAST CHROME DYEINGS ON WOOL AND NYLON SLUBBINGS—This card contains twenty-four matched pairs of dyeings on wool and nylon slubbing, respectively. The colours include greys, dark browns, dark greens, navies, a blue and a red, produced using mixtures of dyes in the majority of instances. In all except two cases where the afterchrome method was used the wool has been dyed using the Metomega Chrome method. The nylon has been dyed afterchrome using 2–4% dichromate and 2% of formic acid (85%) and chroming for 1 hr. at the boil. It is recommended that the dyed nylon be rinsed thoroughly and then soaped at 70–80°C. for 15–20 min. using 1–2 g. soap and 0·5 g. soda ash per litre in order to remove undesirable unreduced dichromate.

SANDOTHRENE AND TETRA DYESTUFFS—This card contains dyeings on cotton cloth in three depths of ninety-four vat dyes including eighty Sandothrene dyes, nine Tetra dyes and five Sandone Blues, the latter being a group intermediate between the sulphur and indigoid vat dyes which can be dyed using a sodium sulphide-hydrosulphite-caustic soda vat or hydrosulphite-caustic soda only. In addition to very comprehensive data concerning fastness properties, alongside each set of patterns is appended a graphical representation of the rate of exhaustion which obtains when dyeing a pale shade. The text includes sections dealing with the behaviour of the dyes during trubenizing, and their influence on the tendering of the fibre on exposure to light after dyeing, during dyeing, and when the dyed material is subsequently treated with bleaching agents containing chlorine.

ALIZARINE MILLING GREEN B—This acid wool dye is recommended for dyeing loose wool, slubbing, yarn and cloth from a neutral or weakly acid dyebath to give bluish greens of good fastness to light and wet treatments. It is also of interest on silk and nylon and for solid dyeings on wool–nylon mixtures and is suitable for Vigoureux printing. Fastness figures on wool include—Light 6, alkaline milling 4, acid milling at 90°C. 4–5.

Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., **68**, *23 (Jan. 1952), and also, together with symbols and the periodicals abstracted, in the annual index*

I—PLANT; MACHINERY; BUILDINGS

Feeding Loose Fibres into Drum Agitators. Roberts (Engineers). *BP* 696,694

The fibres are fed continuously into a pocket, formed, when the door of the drum is closed, by a sheet suspended between the frame of the machine and the free edge of the drum door. They are projected into the drum by the tensioning of the sheet when the drum door is rapidly opened. C. O. C.

Thread-advancing Device. Courtaulds. *BP* 696,365

In order to facilitate cleaning, the tube carrying the treating liquids projects through a gland and can be closed temporarily by moving the tube relatively to the gland. W. G. C.

Wet Treatment of Travelling Yarn. American Viscose Corpn. *BP* 696,486

The yarn is advanced in a helical path, and treatment liquor is applied to it tangentially to the direction in which the turns are rotating. Excess of liquor is thrown off by the centrifugal force developed by the rotation of the helix. C. O. C.

Treating Yarn with Fluid or Vapour. British Nylon Spinners. *BP* 696,483

The yarn is wound onto a hollow foraminous cylinder, from which the fluid or vapour is forced through the package. Sealing members placed in annular grooves on the cylinder seal the end portions of the packages. Each member is made up of (*a*) an inwardly directed flexible wall which can be forced outwardly to touch the adjacent part of the inner wall of the cylinder so as to prevent leakage of the fluid from the ends of the member, and (*b*) a base placed in the annular groove of the cylinder to constrain longitudinal movement of the seal relative to the cylinder. C. O. C.

Continuous Mercerising of Yarn. Dixie Mercerizing Co. *BP* 696,595

A machine for uniformly mercerising a single yarn strand throughout its length. C. O. C.

Rollers for Wet Treatment of Fabrics. Richard Hough. *BP* 696,739

Very satisfactory rollers are cheaply made by rigidly mounting a compressed cylinder of wood fibres on a shaft, the fibres being impregnated with an aqueous liquid before they are mounted on the shaft and then being compressed thereon. C. O. C.

Winch. Colonial Print Works. *USP* 2,624,190

The vat for the dye liquor has oppositely inclined and upwardly diverging end walls and upwardly converging wall sections extending from the central portion of the bottom so as to divide the vat into two sections. A winch is mounted above each section. Each winch has a four-sided cross-section with adjacent sides of unequal lengths. This causes the cloth to be piled in superimposed folds of alternatively long and short lengths. The winches are placed so that the cloth falls on the corresponding end wall, and is guided by that and the bottom of the vat up the corresponding central wall, so that it is relieved of the weight of the remaining folds in the pile and thus is readily withdrawn by the winch. This results in less power being needed to drive the winch and less tension being imparted to the cloth. C. O. C.

Weft-straightening Machine. G. Durrant & Sons. *BP* 697,026

Pairs of rollers are arranged in parallel planes, each alternate pair being in the same plane and staggered relatively to those in the other plane. The mutual angularity of the rollers of each pair can be varied simultaneously in the same manner in their respective planes, so that common tangents between successive rollers are increased at one end while being decreased at the other end. This adjustment is made by an oscillatable shaft moved by an electric motor controllable from a central and/or remote position relative to the machine, the mechanical connection between motor and shaft having limited travel and incorporating a slipping clutch. This enables the operator to stand where he can best view the cloth. C. O. C.

Screen Printing Machine. Meyercord Co. *USP* 2,624,276

Squeegee mechanism by which the squeegee prints only in one direction and not on its return stroke. The clutch mechanism is such that the squeegee and other moving parts of the machine may be stopped with the machine in open position for adjustment without stopping the entire machine. C. O. C.

Raising Machines. B. W. Wood. *BP* 696,437

The cylinder and pile rollers are driven independently of the counter-pile rollers, the speeds of the two separate drives being controllable to vary the relative speeds of each series of rollers. C. O. C.

Machines for Washing, Dry Cleaning, or Garment Dyeing. Isaac Braithwaite & Son Engineers. *BP* 694,575

Washing machines of the type having a cylinder with a horizontal axis rotating in an outer casing with an annular space between are fitted with scoops on the outer periphery of the cylinder. These consist of small curved plates which serve both to scoop up liquid in the peripheral space and to retain it, whence it passes through the perforated cylinder to the inside. J. W. B.

Coating Feed Mechanism for Gate Rolls in a Web-coating Machine. Consolidated Water Power & Paper Co. *BP* 696,397

A coating-feed mechanism for uniformly distributing a liquid coating suspension along the length of a nip between two gate rolls of a coating machine comprises a track disposed substantially parallel to the length of the gate rolls, a carrier positioned on the track, means, e.g. an endless chain or a screw, adjacent and parallel to the track for moving the carrier forwards and backwards, a drive for the carrier mover, and a coating material conduit mounted on the nip to discharge coating material into the nip when the carrier is moved. The drive may be unidirectional. The endless flexible driving mechanism may have two or more parallel passes and means for detachable engagement with the carrier. A lug carried by the chain and a pair of spaced forks on the carrier allow alternate engagement with the lug, the chain lug alternately breaking and making engagement with the forks at the end of each pass forwards and backward. The carrier is held stationary by magnetic means during disengagement and engagement. S. V. S.

Drying by Radiant Heat. FBy. *BP* 696,655

The material is placed on a conveyor belt which may be below and parallel to a number of tubes radiating heat. There are burners at the inlets of the tubes and means for withdrawing the combustion gases from the outlets. C. O. C.

Drier. Ercole Marelli & C. *BP* 696,792

The material is placed in a chamber where a stream of air is directed on to it from a fixed point within the chamber. The stream of air rotates uniformly about the fixed point, e.g. from a rotatable nozzle, at a speed <1 r.p.m., fresh air being introduced into the chamber at the same rate as the moist air is withdrawn. This prevents formation of a crust on the surface of the material being dried. C. O. C.

Drying Machine for Printed or Coated Material E. G. Rice. *USP* 2,624,573

The printed or coated material is carried through a hot-air drying chamber on an endless belt which follows a very long helical path through the chamber. The belt moves at a high speed. This enables drying to be carried out in a relatively limited space without any risk of smudging. C. O. C.

Fabric Drying. Hicking, Pentecost & Co. *BP* 694,499

Tubular knitted fabric is continuously dried by passing upwards over a stretching device comprising a series of connected rings floating within the fabric. The stretching device is surrounded by a box, through which the fabric passes to the take-off rollers. Baffles fitted in an upper compartment of the box around the stretching device conduct hot air into the fabric, the air escaping through the fabric into a lower compartment for collection and reheating. G. E. K.

Hosiery Drying. Mellor Bromley. *BP* 694,509

A circular table on which hosiery forms are vertically mounted rotates within a circular drying chamber. The forms pass through an annular space in which a downward current of hot air is circulated. A section of the chamber is cut away to permit continuous stripping and replenishment of the forms. G. E. K.

Cloth Inspection Machine. Celanese Corpn. of America. *BP* 697,504

Driving mechanism for moving cloth across an inspection board at controlled and constant speed in either direction while maintaining controlled, uniform tension. C. O. C.

Forms for Examining Tubular Fabric and Hosiery. Kilbourn Martin. *BP* 697,573

Garment Presses. R. G. Whitaker. *BP* 697,132

Control of the pressure between back and head is such that from full pressure the press can be adjusted to deliver a change of full pressure for starting and a reduced final pressure to suit the garment being pressed, or to stop the press with a gap or only light contact between head and back when it is desired to finish a garment by steaming with little or no pressure. C. O. C.

Garment Press. Brown & Green. *BP* 697,317

Hat-pressing Apparatus. Hat Corpn. of America. *USP* 2,624,495

A semi-automatic power-operated press which rapidly shapes and presses a hat body, giving uniform pressing at all times. C. O. C.

Cleaning and Sorting Feathers. L. H. Lorch. *BP* 696,877

In a machine where the feathers are carried by a current of air through several chambers, the speed and the quantity of air flowing through the upcast channels of the chambers are both adjustable. This results in better sorting. C. O. C.

Hydraulic Presses. Turner Tanning Machinery Co. *BP* 696,835

A press specially suitable for smooth-plating or embossing leather has its main ram or piston formed or equipped at its inner end (i.e. the end moving within the main cylinder) to cooperate with hydraulic fluid in the cylinder and function as a dashpot to dampen the press-opening movement. C. O. C.

Fuel Efficiency in the Dyeing and Finishing Industries (VIII p. 470).

II—WATER AND EFFLUENTS

Water Conditioning for the Textile Plant. E. Nordell. *Amer. Dyestuff Rep.*, **42**, P 371–P 378 (8 June 1953).

A survey is presented of waters and their impurities, with an account of different methods of softening and a description of precipitating and exchanging, etc. plants used. J. W. B.

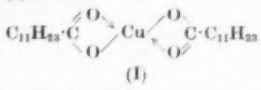
III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Chemical Utilisation of Fats and Oils. I—Preparation of Aralkyl Ketones and Hydrocarbons. R. D. Englert, R. M. Silverstein, and L. M. Richards. *J. Amer. Oil Chem. Soc.*, **30**, 337–339 (Aug. 1953).

A number of straight-chain aralkyl detergent intermediates were prepared from fatty acids. The most satisfactory method of preparation involved a Friedel–Crafts condensation of the acyl chloride with the aromatic followed by catalytic hydrogenation. P. G. M.

Magnetic Properties and Structure of Copper Laurate. A. Gilmour and R. C. Pink. *J.C.S.*, 2198–2201 (Aug. 1953).

Although early measurements of the magnetic susceptibility of copper soaps revealed no abnormal behaviour, subsequent measurements have indicated that the magnetic moment of the copper in these compounds is anomalously low. This is now confirmed, and it is shown further that copper laurate becomes anti-ferromagnetic at low temperatures. This is explained by assuming the existence of strong quantum-mechanical exchange forces between the copper atoms. A covalent chelate structure (I) for copper laurate is suggested, based on magnetic and chemical evidence provided by measurements on copper laurate and its co-ordination compounds with ammonia and pyridine.



(I)

H. H. H.

Fat-based Textile Auxiliaries. L. M. Miller. *Amer. Dyestuff Rep.*, **42**, P 435–P 440 (6 July 1953).

A survey of textile auxiliaries based on long-chain fatty acids and alcohols. J. W. B.

Methoxyl Content of Gum Tragacanth. K. Selby. *J.C.S.*, 2504 (Aug. 1953).

Based on changes in the carboxyl and methoxyl contents of alkaline solutions of the gum on storage for several days in a refrigerator, the whole of the methoxyl content is shown to occur in the form of methyl ester (cf. the claim of James and Smith, *J.C.S.*, 739 (1945) that the methoxyl content (3·8%) is of an etheric nature and that it is unchanged by aq. NaOH). H. H. H.

Structure of Karaya Gum (*Cochlospermum gossypium*). E. L. Hirst and S. Dunstan. *J.C.S.*, 2332–2337 (Aug. 1953).

This gum is a partly acetylated acidic polysaccharide which, on hydrolysis, gives equimolecular proportions of L-rhamnose, D-galactose, and D-galacturonic acid, together with traces of a ketohexose. On partial hydrolysis, it gives these together with 2-D-galacturonosyl-L-rhamnose, 4-D-galacturonosyl-D-galactose, and unidentified oligosaccharides of high uronic acid content. The homogeneous methylated gum, on hydrolysis, gives 2:3:4-trimethyl (1 part), 3:4-dimethyl (1 part), and 3-methyl rhamnose (1 part), 2:3:4:6-tetramethyl (2 parts), 2:3:6-trimethyl (1 part), and 2:6-dimethyl galactose (ca. 1%), and a mixture of methylated uronic acids. The gum possesses a highly branched structure. H. H. H.

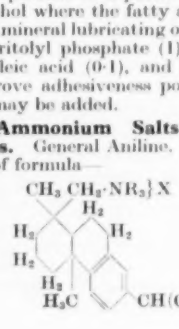
PATENTS

Textile Machinery Lubricant. Standard Oil Development Co. *BP* 694,425

A lubricating composition suitable for high-speed machines, e.g. knitting and spinning machines, which minimises stain formation on the fabric, consists essentially of a well refined mineral-base oil having a viscosity of 50 S.S.U. at 100°F. to 75 S.S.U. at 210°F., together with a small proportion of an oil-soluble ester of an acid of phosphorus in which each ester group contains 6–24 C, a small proportion of an alkylated dicarboxylic acid in which the alkyl group contains 12–24 C or a mono- or di-alkyl ester of such acid or a mono- or di-alkyl ester of dicarboxylic acid in which each ester group contains 8–24 C, and a small proportion of a partial fatty-acid ester of a polyhydric alcohol where the fatty acid contains 12–24 C. In one example mineral lubricating oil (98·4 parts by wt.) is blended with tritolyl phosphate (1), a C_{12}–C_{16} alkylated succinic or maleic acid (0·1), and sorbitan mono-oleate (0·5). To improve adhesiveness polyisobutylene of mol. wt. 5000 (0·5) may be added. J. W. B.

Quaternary Ammonium Salts—Emulsifiers and Detergents. General Aniline. *USP* 2,623,870

Compounds of formula—



(X = anion; R = Alk of 1–4 C, β-hydroxethyl, or polyethylene ether radicals containing >1 oxyethylene radicals) have outstanding emulsifying power for water-insoluble hydrocarbons and detergent properties in aqueous solution. C. O. C.

Tetraborate Detergent. General Aniline. *USP* 2,623,856

Alkali-metal tetraborates are readily converted into low-density hollow beads by conventional spray drying. They can be mixed with 40% of surface-active liquid non-ionic polyglycol ether compounds while still remaining dry and free flowing. C. O. C.

Concentrated Soap Solutions of Low Viscosity. Celanese Corpn. of America. *BP* 697,510

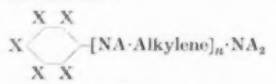
The viscosity of soap solutions, particularly of sodium oleate, can be much reduced by adding a small quantity of an alkyl oleate (Alk of 3–10 C), or of a mineral oil and a non-saponaceous emulsifying agent. C. O. C.

Alkylthiophensulphonate Detergents. Universal Oil Products Co. *USP* 2,624,742

The products obtained by alkylating thiophen with a mono-olefinic alkylating agent of 9–18 C, separating a desired alkylthiophen from the reaction mixture, and then sulphonating and neutralising the alkylthiophen, are detergents which resist hard water. C. O. C.

Chelating Compounds for Metal Ions. F. C. Bersworth. *USP* 2,624,756

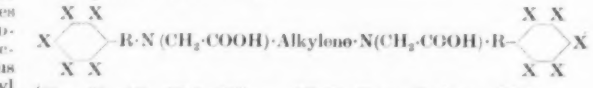
Compounds of formula—



(X = H, OH, Alk, Alk·O, or Hal, at least one but not all being Hal; A = CH_2·COOH or CH_2·CH_2·COOH; n = 1–5; Alkylene = ethylene, propylene, or trimethylene) are water-soluble germicidal metal-ion-chelating compounds active over a wide pH range.

USP 2,624,757

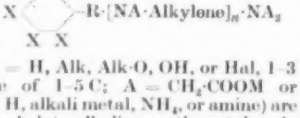
Compounds of formula—



(X = H, Alk, Hal, OH, or Alk·O; R = alkylene which may contain ether linkages) are soluble in both water and organic solvents. In both solutions they have strong chelating properties towards heavy-metal ions and have bactericidal and fungicidal properties in aqueous solution.

USP 2,624,758

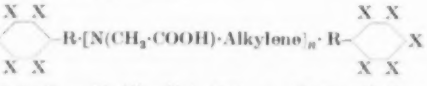
Compounds of formula—



(n = 1–5; X = H, Alk, Alk·O, OH, or Hal, 1–3 being Hal; R = alkylene of 1–5 C; A = CH_2·COOM or CH_2·CH_2·COOM; M = H, alkali metal, NH_4, or amine) are germicidal agents which chelate alkaline-earth-metal and magnesium ions in aqueous solution.

USP 2,624,759

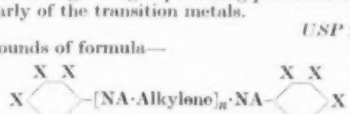
Compounds of formula—



(n = 3–6; X = H, Alk, Hal, OH, or Alk·O; Alkylene = ethylene, propylene, or trimethylene; R = $[CH_2]_m$; m = 1–5) are highly water-soluble bactericides and fungicides having strong sequestering power for metal ions, particularly of the transition metals.

USP 2,624,760

Compounds of formula—



(X = H, Alk, Alk·O, OH, and Hal, at least one being Hal; n = 1–5; A = CH_2·COOH or a homologue thereof;

Alkylene = ethylene, propylene, or trimethylene) are bactericidal metal-ion-chelating agents in aqueous solution. C. O. C.

Viscous Urea Formaldehyde Resin Compositions. DuP. *USP* 2,625,524

Clear, liquid, permanently stable, unpolymerised urea–formaldehyde compositions of high resin-forming content, formed by keeping the formaldehyde above 45° c. from the time it is formed until it is converted into polyhydroxymethylurea, on being mixed with urea yield a stable partly polymerised, viscous, liquid, urea–formaldehyde composition suitable for the production of chintz and similar finishes. C. O. C.

Aqueous Emulsions of Organic Silicates—Non-slip Finish. Monsanto. *BP* 697,451

Aqueous emulsions of organic silicates containing a protective colloid or butyl laurate confer a non-slip finish which has good resistance to laundering. C. O. C.

Addition Products of an Amine and Sulphone-activated Ethylenic Compounds. American Viscose Corpn. *USP* 2,623,035

The products obtained by reaction and/or condensation of urea with a sulphone-activated ethylenic compound, e.g. divinyl sulphone, have a wide variety of uses. Thus the monomeric products are useful as textile assistants, while the polymerised products are useful in coatings and mouldings and for drawing into fibres or films. C. O. C.

Stabilised Aqueous Hydrogen Peroxide. Buffalo Electro-Chemical Co. *USP* 2,624,655

Aqueous hydrogen peroxide of any strength is stabilised by addition of a little of one or more pyridine carboxylic acids. C. O. C.

Stabilised Papain Composition. Sterling Drug. *BP* 696,986

A stable papain composition is obtained by treating the fresh latex with 0·1–3·0% by weight of sodium bisulphate before drying. Better results are obtained if small amounts of sodium bisulphite and a phenolic compound are also present. The resulting compositions retain their proteolytic activity unimpaired after 2–4 years' storage, whereas unstabilised compositions show a 25–50% loss in activity when stored for 2 years. C. O. C.

Catalysts for Acid-curing Thermosetting Aminoplast Condensates. Monsanto. *BP* 697,479

A water-soluble salt of (*a*) an aliphatic primary or secondary monoamine which may contain 1–4 OH groups, or a primary or secondary monoamine of a *cyclo*paraffin or an alkyl*cyclo*paraffin, and (*b*) a strong mineral acid, aliphatic mono-(or di)carboxylic acid, together with <5% (on the chemical equivalent of the amine combined in the salt) of a water-soluble primary or secondary monoamine, gives good stabilisation at room temperature with effective curing on heating. C. O. C.

Germicidal Complex Compounds for Application to Textiles, Rubber, Plastics, etc. Permachem Corpn. *BP* 696 912

A mixture of a silver salt and a monohydroxyalkylamine reacts to form a complex which usually yields a clear aqueous solution having many times the germicidal power of phenol. C. O. C.

Polyvinyl Alcohol—Use as a Fibre and in Textile Finishes (VI p. 469).
Protein Solutions (VI p. 469).
Propyl Dixylyl Phosphate (XIII p. 477).

IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

Vapour Pressure and Lattice Energy of some Aromatic Ring Compounds. R. S. Bradley and T. G. Cleasby. *J.C.S.*, 1690–1692 (June 1953).

The vapour pressures, lattice energies, and entropies of vaporisation are determined by an effusion method for naphthalene, anthracene, phenanthrene, fluorene, diphenyl, and pyrene. For the three similar compounds—benzene, naphthalene, and anthracene—the lattice energy per C-atom is very nearly the same, viz. 1·78, 1·73, and 1·74 kg.cal. respectively; for phenanthrene and pyrene the values are 2·07 and 2·25 kg.cal.; and for diphenyl and fluorene 1·95 and 1·98 kg.cal. The data all correspond to the respective structures of these hydrocarbons. H. H. H.

Hyperconjugation. I—Conjugation and Hyperconjugation Energies. C. A. Coulson and V. A. Crawford, *J.C.S.*, 2052–2058 (July 1953).

The calculations of Mulliken, Rieke, and Brown and of Roberts and Skinner of the hyperconjugation energies and bond orders for a number of methyl-benzenes and -ethylenes are improved and extended. It is found that the hyperconjugation energy per CH_3 group is constant, and that the strengthening of single and double bonds by hyperconjugation is less than previous estimates, the total contribution of third-order conjugation to the bond energies of both single and double bonds being only ~1·6 kg.cal. H. H. H.

Hyperconjugation. II—Charge Distributions in Propylene and some Methylbenzenes. V. A. Crawford, *J.C.S.*, 2058–2061 (July 1953.)

The π-electron densities in propylene and some polymethylbenzenes are computed theoretically and correlated with their substitutional reactivity. H. H. H.

Hyperconjugation. III—Bathochromic Shifts resulting from Methyl Substitution in Conjugated Systems. V. A. Crawford, *J.C.S.*, 2061–2065 (July 1953).

From a molecular-orbital calculation of the frequency of absorption of methyl-substituted ethylenes and benzenes, it is concluded, in agreement with Mulliken, that the red shifts in the spectra result chiefly from hyperconjugation. H. H. H.

Lipoid–Water Partition Coefficients of some Aromatic Bases. J. Cymerman-Craig and A. A. Diamantis. *J.C.S.*, 1619–1621 (May 1953).

The partition coefficients between liquid paraffin and aqueous phosphate buffer (pH 7·2) are reported for 26 aromatic bases of pK_a values ranging from 3 to 10. They were determined by a spectrophotometric estimation of the organic base in the aqueous phase. Lipophilic character is found to increase with molecular complexity. H. H. H.

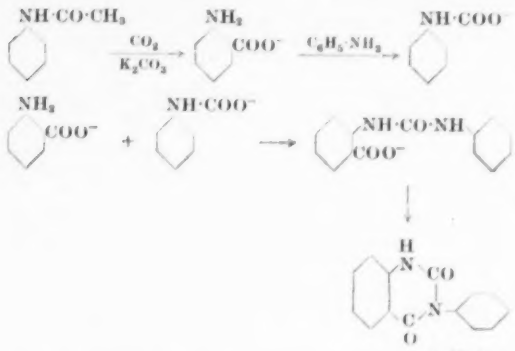
Oxidations with Phenyl Iodosoacetate. II—Oxidation of Primary Aromatic Amines. K. H. Pausacker. *J.C.S.*, 1989–1990 (July 1953).

Phenyl iodosoacetate oxidises the following primary aromatic amines in benzene solution to give—Azo Compounds (% yields in parentheses) from aniline (95), *o*- (42), *m*- (56), and *p*-toluidine (6), *o*- (39), *m* (66), and *p*-chloroaniline (55), *o*- (3) and *p*-anisidine (5), *p*-phenetidine (7), *m*- (65) and *p*-nitroaniline (53), and *α*-naphthylamine (3); Benzofurazan Oxides from *o*-nitro- (81), 4-methyl- (96), 4-chloro-2-nitro- (91), and 2:4-dinitroaniline (94); and 1:2:6:7-Dibenzophenazine (28) from *β*-naphthylamine together with a product $C_{25}H_{20}O_5N_2$ which has a green metallic lustre and gives a deep blue solution in nitrobenzene. The characteristic colour reactions when ca. 1 mg. of amine is added to 2 ml. of 1% phenyl iodosoacetate in glacial acetic acid are recorded. H. H. H.

Introduction of the Carboxyl Group into Aromatic Amines. A. I. Kizber and A. S. Glagoleva. *J. Gen. Chem. U.S.S.R.*, **23**, 1028–1034 (June 1953).

Heating of an aromatic amine under pressure with CO_2 and K_2CO_3 yields small amounts of *o*- and *p*-aminocarboxylic acids, but with an acyl derivative of the amine (an acetamido or benzamido cpd., a *s*-diarylurea, or an aryl *iso*cyanate) the same reaction (185–260° c.) gives these acids in good yield (based on net amount of amine consumed)—they are obtained partly in the form of a 3-aryltetrahydroquinazoline-2:4-dione, which may be split by caustic alkali into the amino acid and the original amine (see abstract of Dokunikhin and Gueva, J.S.D.C., **69**, 456 (Nov. 1953)). A third reaction product is an *N*-arylcarbamic acid, and evidence given indicates that it is an intermediate product in the formation of the tetrahydroquinazoline deriv. The reaction is carried out on acyl derivatives of aniline *m*- and *p*-toluidine *m*-chloroaniline,

and *α*-naphthylamine. An example of its supposed course is—



A. E. S.

Interaction of Sulphur Monochloride and some Aromatic Amines. R. D. Shingte, A. V. Rege, D. G. Pishavikar, and S. V. Shah. *J. Univ. Bombay*, Sectn. A, **20**, Pt. 3 (*Science No.*, No. 30), 89–92 (1951): *Chem. Abs.*, **47**, 7454 (10 Aug. 1953).

Acetanilide (10 g.) in 40 c.c. cold acetic acid refluxed for 1 hr. with S_2Cl_2 (5 g.) in 5 g. acetic acid, on dilution with water and filtration, yielded $(p\text{-}CH_3\cdot CO\cdot NH\cdot C_6H_4)_2S_2$ (I) as golden-yellow plates. I with dil. KOH yielded benzidine, which when diazotised and coupled with *β*-naphthol gave a red dye. Anthranilic acid (10 g.) in 40 c.c. acetic acid and S_2Cl_2 (10 g.) in 10 g. acetic acid gave $[3{:}4\text{-}COOH(NH_2)C_6H_3]_2S_2$, which when diazotised and coupled with *β*-naphthol gave a red dye. C. O. C.

Catalytic Alkylation of Aniline with Ethanol. M. B. Turova-Polyak, N. V. Borunova, and E. S. Semenova. *J. Gen. Chem. U.S.S.R.*, **23**, 1024–1027 (June 1953).

Aniline is catalytically ethylated in the vapour phase to ethylaniline by means of ethanol; bentonite and activated charcoal impregnated with phosphoric acid are used as catalysts. The best yields are obtained at a molar ratio (aniline : ethanol) of 1 : 2, and the optimum conditions are—bentonite, 350° c., space velocity 0·15 (87% yield on aniline); or charcoal–phosphoric acid, 275°c., space velocity 0·3 (59% yield). A. E. S.

Preparation of some *m*- and *p*-Alkylanilines. R. Long and K. Schofield. *J.C.S.*, 2066–2068 (July 1953).

Catalytic reduction, with Pd–$BaSO_4$ as catalyst and $HClO_4$ as activator, of *m*- and *p*-acetamidophenyl alkyl ketones in acetic acid is found to afford high yields of *m*- and *p*-alkylanilines. Direct hydrogenation of the nitro- or amino- ketones under the same conditions gives only small amounts of alkylanilines and large quantities of high-boiling oils. H. H. H.

3-Nitro-2-Naphthylamine. E. R. Ward and T. M. Coulson. *Chem. and Ind.*, 542–543 (30 May 1953).

Preparation of 3-nitro-2-naphthylamine by the route aceto-3-nitro-1-naphthalide→aceto-2:3-dinitro-1-naphthalide → 2:3-dinitronaphthalene → 3-nitro-2-naphthylamine has led to contradictory results. It is now shown that nitration of aceto-3-nitro-1-naphthalide gives a mixture of aceto-3:5- and -3:8-dinitro-1-naphthalides; these are identified by hydrolysis, separation of the two amines, and subsequently deamination to 1:7- and 1:6-dinitronaphthalenes. The substance used previously for the preparation of "3-nitro-2-naphthylamine" was, therefore, actually a mixture of 1:6- and 1:7-dinitronaphthalenes, which reduce to a varying mixture of 5- and 8-nitro-2-naphthylamines. J. W. B.

Hydrophilic Derivatives of 4-Aminodiphenyl. J. N. Baxter, J. Cymerman-Craig, and W. S. Gilbert. *J.C.S.*, 2400–2403 (Aug. 1953).

The preparations of 2-*p*-diphenylylaminoethanol and 3-*p*-diphenylylaminopropane-1:2-diol are described. Basic strengths and oil–water partition coefficients at pH 7·2 of these and related compounds are determined and discussed. H. H. H.

3:6-Disubstituted Fluorenes. I—Attempted Introduction of a 6:6′-Methylene Bridge directly into 3:3′-Diaminodiphenyl and some of its Derivatives. A. Barker and C. C. Barker. *J.C.S.*, 2034–2036 (July 1953).

Earlier claims to have prepared 3:6-bis-dimethylamino-9-phenylfluorene from 3:3′-bis-dimethylaminodiphenyl and benzylidene chloride, or fluorene derivatives by condensing HCHO with either this compound, 3:3′-diaminodiphenyl, or 3:3′-bis-dimethylamino-4:4′-dimethyldiphenyl, are refuted. Formaldehyde appears to attack such amines only at a vacant 4-position. H. H. H.

Syntheses of some Styryl and Cyanostilbene Derivatives. A. Schonne, E. Braye, and A. Bruylants. *Bull. Soc. chim. Belg.*, **62**, 155–171 (March–April 1953).

Coloured styryl and cyanostilbene derivatives of general formulæ $R\cdot C(CN){:}CH\cdot C_6H_5$ and $R\cdot C(CN){:}CH\cdot C_6H_4\cdot N(CH_3)_2(p)$ are obtained by the Meyer–Frost (*Annalen*, **250**, 157 (1888)) condensation of benzaldehyde and of *p*-dimethylaminobenzaldehyde with nitriles (9 examples) of the type $R\cdot CH_2\cdot CN$ (R = CN, COOH, C_6H_5, or $C_6H_4\cdot X$). Full details of preparation are included. 21 References. H. H. H.

Synthesis of some *N*-Acyl Derivatives of 4:4′-Diaminostilbene-2:2′-disulphonic Acid. B. M. Bogoslovsky. *J. Appl. Chem. U.S.S.R.*, **26**, 225–227 (Feb. 1953).

4:4′-Diaminostilbene-2:2′-disulphonic acid ($H_2N\cdot X\cdot NH_2$) is acylated with (*a*) benzoyl chloride, (*b*) nicotinoyl chloride, and (*c*) malonyl chloride, the products being (*a*) $C_7H_5\cdot CO\cdot NH\cdot X\cdot NH\cdot CO\cdot C_7H_5$, (*b*) $C_5H_4N\cdot CO\cdot NH\cdot X\cdot NH_2$, and (*c*) $H_2N\cdot X\cdot NH\cdot CO\cdot CH_2\cdot CO\cdot NH\cdot X\cdot NH_2$. The Skraup reaction, like reactions (*b*) and (*c*), proceeds at one end of the mol. only, giving the expected quinoline derivative. The substances obtained are of interest in the preparation of dyes. A. E. S.

3:4-, 3:5-, and 3:6-Dimethylcatechol. W. Baker, H. F. Bondy, J. Gumb, and D. Miles. *J.C.S.*, 1615–1619 (May 1953).

3:5- and 3:6-Dimethylcatechol are isolated from a coal tar produced by low-temperature carbonisation, and, together with the 3:4-isomeride, are synthesised by oxidation with H_2O_2 in aqueous tetramethylammonium hydroxide of the relevant 2-hydroxydimethyl-acetophenone or -benzaldehyde. Synthetic 3:5- and 3:6-dimethylcatechol do not give a red colour in aq. NaOH in contrast to the 3:4-isomeride, whereas the presence of a resorcinol produces a crimson with the 3:5- but not with the 3:6-dimethylcatechol. It would appear that a colour reaction occurs only with a catechol having a CH_2 group *para* to one or other of the OH groups. H. H. H.

Syntheses with the aid of γ-Picolyllithium. I—Preparation of 4-Alkylpyridines. J. P. Wibaut and J. W. Hey. *Rec. Trav. chim.*, **72**, 513–521 (June 1953).

Synthesis of 3:5-Dinitropyridine. E. Plažek. *Rec. Trav. chim.*, **72**, 569–575 (June 1953).

Reactions of Methazonic Acid. III—Novel Reaction of 3-Aminolepidine. D. W. Ockenden and K. Schofield. *J.C.S.*, 1915–1919 (June 1953).

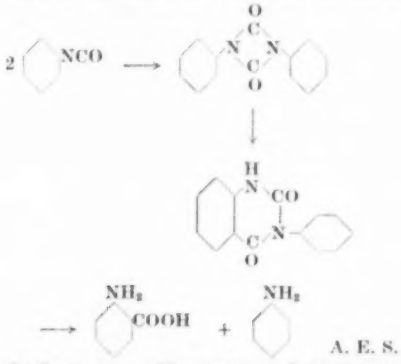
3-Aminolepidine (I) reacts with HNO_2 (2 mol.) to give 1:2:3:9-tetra-azaphenanthrene 3-oxide, which is converted by acid into 3-azidoquinoline-4-aldehyde, and thence by heat into quinoline(3′:4′-3:4) sydnone. Diazotisation of I in 2 N-H_2SO_4 gives some 3*H*-1:2:6-triaza-4:5-benzindene, but in conc. HCl an initial deep violet colour is observed which does not occur in H_2SO_4 of similar concn., and which remains unexplained. 3-Amino-6-chlorolepidine gives similar reaction. The *N*-nitroso derivative of 3-acetamidolepidine is converted by hot benzene into 3*H*-1:2:6-triaza-4:5-benzindene. H. H. H.

Some 6-Alkyl-5- and 7-Alkyl-8-aminoquinolines. R. Long and K. Schofield. *J.C.S.*, 2350–2354 (Aug. 1953).

Organic *iso*Cyanates. I—Action of Aluminium Chloride on *iso*Cyanates of the Benzene Series. N. S. Dokunikhin and L. A. Gaeva. *J. Gen. Chem. U.S.S.R.*, **23**, 606–610 (April 1953).

An *iso*cyanate of the benzene series or its dimer (which is formed in the course of the reaction if monomer is used) on fusion with a mixture of aluminium and sodium

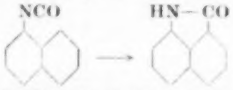
chlorides yields a 3-aryltetrahydroquinazoline-2:4-dione, which is converted by alkali into a mixture of the arylamine and the *o*-aminobenzoic acid. The reaction thus provides a means of introducing an *o*-carboxyl into an aromatic amine. Compounds containing the following aryl radicals are investigated—phenyl, *o*-, *m*-, and *p*-tolyl, 2:4-xylyl, *o*-, *m*-, and *p*-chlorophenyl, 2:5-dichlorophenyl, and *p*-diphenylyl. For phenyl *iso*cyanate the reaction is—



A. E. S.

Organic *iso*Cyanates. II—Action of Aluminium Chloride on *iso*Cyanates of the Naphthalene Series. N. S. Dokunikhin and I. D. Pletneva. *J. Gen. Chem. U.S.S.R.*, **23**, 798–800 (May 1953).

1-Naphthyl *iso*cyanate and its derivatives (4-chloro- and 2-methyl were investigated), unlike *iso*cyanates of the benzene series (see previous abstract), do not form derivatives of tetrahydroquinazoline when treated with aluminium chloride, but yield naphthastyrils, e.g.—



A. E. S.

Structure and Chemical Properties of Dyes in Relation to their Use. T. Vickerstaff. J.S.D.C., **69**, 279–282 (Aug. 1953).

The Search for New Dyes in relation to Modern Developments in the Textile Field. T. Holbro. J.S.D.C., **69**, 233–240 (July 1953).

Disperse Dyes. C. M. Whittaker. J.S.D.C., **69**, 205 (June 1953).

Thermal Decomposition of Hydrazobenzene, studied by the Use of Nitrogen Isotopes. P. F. Holt and B. P. Hughes. *J.C.S.*, 1666–1669 (June 1953).

A modified Dumas procedure is described which is a satisfactory alternative to the Rittenberg method for the preparation of nitrogen for mass-spectrographic analysis. When decomposed by the new process, [^{15}N]azobenzene, labelled in one position, gives nitrogen without complete random pairing of the atoms. The formation of azobenzene by the thermal decomposition of hydrazobenzene does not involve separation of the nitrogen atoms, and the mechanisms proposed by Stieglitz and Curme are, therefore, invalid. H. H. H.

N—N, N=N, and N≡N Bond-stretching Frequencies. W. J. O. Thomas. *Chem. and Ind.*, 567 (6 June 1953).

The difficulty of assigning a well-defined frequency to a single bond is discussed with reference to the azo group, the azide ion, and the molecules HNCO and HNCS.

J. W. B.

Aromatic Azo Compounds. I—Oxidation of *cis*- and *trans*-Azobenzene. G. M. Badger, R. G. Buttery, and G. E. Lewis. *J.C.S.*, 2143–2147 (July 1953).

Oxidations by perbenzoic acid of *cis*-azobenzene, *cis*-*m*-azotoluene, and *cis*-*p*-azotoluene to the respective *cis*-azoxy compounds are described, *trans*-azobenzene gives *trans*-azoxybenzene in the dark, but some *cis*-azoxybenzene is obtained in sunlight. Reduction of *cis*-azoxybenzene with $LiAlH_4$ gives *trans*-azobenzene. The rate of oxidation of *cis*- is much greater than that of *trans*-azobenzene, and indicates that the electron density at the azo linkage in the *cis* is much greater than that in the *trans* isomer. H. H. H.

Aromatic Azo Compounds. II—Oxidation of Substituted Azobenzenes. G. M. Badger and G. E. Lewis. *J.C.S.*, 2147–2150 (July 1953).

The rates of oxidation with perbenzoic acid of various substituted azobenzenes are determined at three temperatures, and the energies of activation are calculated. All *meta*- and *para*-substituted compounds are found to have the same entropy of activation. Electron-releasing and -attracting substituents respectively decrease and increase the heat of activation and its free energy. The effects of two substituents are accurately additive, and a linear relationship exists between the rate constants and the Hammett σ constants for the various substituents, and is of value in determining the relative electron densities around the azo nitrogen atoms in carcinogenic compounds.

H. H. H.

Aromatic Azo Compounds. III—Oxidation of Azonaphthalenes and Phenylazonaphthalenes. G. M. Badger and G. E. Lewis. *J.C.S.*, 2151–2155 (July 1953).

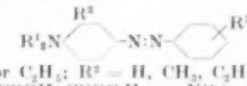
The rates of oxidation of the two phenylazonaphthalenes and of the three azonaphthalenes with perbenzoic acid in benzene at 15°, 20°, 25°, and 30°C. are reported, the energies of activation calculated, and the results interpreted in terms of the extent of conjugation of the azo group with the ring system, and of steric hindrance. It is also concluded that the carcinogenic substance, 2:2′-azonaphthalene, is *not* closely related to the carcinogenic aminoazo compounds. H. H. H.

Aromatic Azo Compounds. IV—Absorption Spectra of Azo and Azoxy Compounds. G. M. Badger and R. G. Buttery. *J.C.S.*, 2156–2158 (July 1953).

It is found that the *K*-absorption band in the spectra of the azonaphthalenes and phenylazonaphthalenes can be correlated with the degree of conjugation. Further, there is a bathochromic shift, in most cases, in passing from the azo to the azoxy compound, although with 1:1′-azoxynaphthalene and *oo*′-azoxytoluene there is a pronounced hypsochromic shift, which is probably due to steric hindrance to coplanarity. H. H. H.

Auxochromic Effect of the Dimethylamino Group and its Coplanarity with the Benzene Ring—II. A. I. Kiprianov and I. N. Zhmurova. *J. Gen. Chem. U.S.S.R.*, **23**, 626–634 (April 1953).

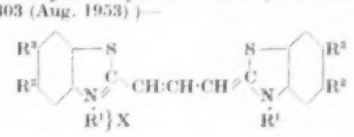
The effect on the spectral characteristics of a dye of a disturbance, due to steric hindrance, in the coplanarity of a ring system of the dye molecule and an attached dimethylamino group—which has already been examined for cyanine dyes (see J.S.D.C., **69**, 33 (Jan.), 303 (Aug. 1953))—is now examined for seventeen azo dyes (eight of them new) of formula—



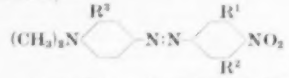
(R^1 = CH_3 or C_2H_5; R^2 = H, CH_3, C_2H_5, or $(CH_3)_2CH$; R^3 = SO_3H, COOH, $COOC_2H_5$, or NO_2). Introduction of a substituent *ortho* to dimethylamino leads to a shift towards the short waves in the absorption max. and to a reduction in the extinction coefficient. In an acid medium all the compounds undergo an indicator change, the absorption max. moving towards the long waves; but, whereas in absence of an *ortho* substituent the extinction coefficient increases considerably in this change, all the *ortho*-substituted compounds (except *o*-methyl-substituted Methyl Red) show greatly reduced absorption, which is attributed to the occurrence of salt formation at the dimethylamino group (with complete loss of its auxochromic effect), instead of at the azo group. A. E. S.

Coplanarity of the Nitro Group with the Benzene Ring and its Effect on the Colour of a Dye. A. I. Kiprianov and I. N. Zhmurova. *J. Gen. Chem. U.S.S.R.*, **23**, 874–879 (May 1953).

An examination is made of the spectral characteristics of eight thiacyanine dyes of formula (cf. J.S.D.C., **69**, 33 (Jan.), 303 (Aug. 1953))—



(R^1 = CH_3 or C_2H_5; R^2 = H, CH_3, C_2H_5, or $C(CH_3)_3$; R^3 = H or NO_2; X = anion) and five azo dyes of formula (cf. above abstract) —



(R^1, R^2, R^3 = H or CH_3). Introduction into such dyes, containing NO_2 and having no *ortho* substituents, of one *o*-CH_3 or *o*-C_2H_5 has little effect on the absorption max., but introduction of one *o*-$C(CH_3)_3$ or of two *o*-CH_3 leads to a hypsochromic shift. Thus, steric deformations of NO_2 and of $N(CH_3)_2$ (*loc. cit.*) are similar in their effects, but NO_2, being smaller, is not greatly affected by a single *o*-CH_3 or *o*-C_2H_5. A. E. S.

Effect of Solvents on the Tautomeric Equilibria of 4-Arylazo-1-naphthols and the *ortho*-Effect. A. Burawoy and A. R. Thompson. *J.C.S.*, 1443–1447 (May 1953).

The differences in the effects of solvents and of *ortho*-substituents on the tautomeric equilibria of 4-arylazo-1- and 1-arylazo-2-naphthols, as observed in their electronic spectra, are discussed. These originate mainly in the ability of both tautomers of the 4-arylazo-1-naphthols (in contrast to those of the 1-arylazo-2-naphthols) to form external hydrogen bonds with suitable solvents. The inhibition of external hydrogen-bond formation by *ortho*-substituents in the phenylhydrazone tautomers is mainly responsible for their surprisingly low concentrations in solvents such as water, ethyl alcohol, and acetic acid. H. H. H.

Chromatography of Synthetic Dyes. L. Roosens. *Industrie chim. belge*, **17**, 211–216, 339–353 (1952): *Chem. Abs.*, **47**, 7781 (10 Aug. 1953).

Consideration of absorption data obtained by previous workers suggests the following general rules— (1) the greater the number of azo groups, the stronger the absorption; (2) OH in the 2-position has a greater effect on the naphthalene nucleus than it has when in the 1-position; (3) *o*-hydroxyazo dyes absorb more strongly than *p*-hydroxyazo dyes; (4) dyes containing a thio or a hydroxyl group are more strongly absorbed than dyes free from those groups; (5) aminonaphtholsulphonic acid with dyes having a 2-amino group have stronger absorption than with a 1-amino group; (6) the longer the wavelength at which the compound is absorbed, the higher is the absorption zone, in general, in the column; (7) the greater the mol. wt., the stronger the adhesion; (8) dyes containing halogen atoms absorb more strongly than similar dyes containing a halogen substituent [*sic*]; and (9) the more double bonds in the molecule, the stronger the absorption. 191 references. C. O. C.

Action of Copper on *o*-Hydroxyazo Dyes in Presence of Oxidising Agents. H. Pfitzner. *Angew. Chem.*, **64**, 397 (1952); *Chem. Abs.*, **47**, 8029 (25 Aug. 1953).

A new method for introducing OH groups in the *ortho* position of aromatic azo compounds or amines. C. O. C.

Stability Constants for some Metal Chelates of 2-*p*-Dimethylaminophenylazopyridine. I. M. Klotz and W.-C. L. Ming. *J. Amer. Chem. Soc.*, **75**, 4159–4162 (5 Sept. 1953).

Stability constants for the 1 : 1 chelate of 2-*p*-dimethylaminophenylazopyridine with metals at 25°C., pH 6, and ionic strength 0·15 fall in the order Cu > Hg > Ni > Co > Zn > Mn > Ca > Mg. Hydrolytic effects play a dominant rôle in the equilibria of the Cu and Hg chelates. Variation in temperature or ionic strength has only slight effect. C. O. C.

Studies of Metallised Dye Complexes. II— Copper (II) Complexes with *o*-Carboxyphenylazo-β-naphthol and *o*-Carboxyphenylazo-β-naphthylamine. H. B. Jonassen and J. S. Wilson. *J. Amer. Chem. Soc.*, **75**, 4201–4204 (5 Sept. 1953).

The above two azo dyes react with Cu in a 1 : 1 ratio, apparently by displacement of a hydrogen ion from each *ortho*-substituted group of the dyes. In the case of the copper(II)–*o*-carboxyphenylazo-β-naphthol a monohydroxy addition compound is formed and in the case of the copper(II)–2-carboxyphenylazo-β-naphthylamine a dihydroxy complex results. C. O. C.

Furoyl Azo Dyes. VII— Absorption Spectra of Mono- and Bis-azoacylacetarylides. R. Andrisano. *Ann. Chim.* (Rome), **41**, 545–552 (1951): *Chem. Abs.*, **47**, 8373 (25 Aug. 1953).

Wavelength and extinction coefficient at maximum absorption of solutions in ethyl alcohol or nitrobenzene are given for the following: R·CO·CH(CO·NH·Ar)·N:N·C_6H_5, R·CO·CH(CO·NH·C_6H_4)·N:N·Ar, and [R·CO·CH(CO·NH·Ar)·N:N·C_6H_4]$_2$ when R = CH_3, C_4H_3O, CH_3·C_4H_2O, and Ar = C_6H_5, *o*- and *p*-CH_3·C_6H_4, *o*- and *p*-CH_3·O·C_6H_4, *p*-C_2H_5·O·C_6H_4, *m*- and *p*-Cl·C_6H_4, *p*-Br·C_6H_4, and α- and β-$C_{10}H_7$; also when R = C_6H_5 and Ar = C_6H_5, *p*-CH_3·C_6H_4, *p*-CH_3·O·C_6H_4, *p*-C_2H_5·O·C_6H_4, and *p*-Cl·C_6H_4. The order of bathochromic effects of the R groups is CH_3 < C_6H_5 < C_4H_3O < CH_3·C_4H_2O. C. O. C.

Direct Cotton Dyes derived from Cyanuric Chloride I— Synthesis of Green Dyes and their Properties. H. Iida. *J. Chem. Soc. Japan, Ind. Chem. Sectn.*, **54**, 775–777 (1951): *Chem. Abs.*, **47**, 7781 (10 Aug 1953).

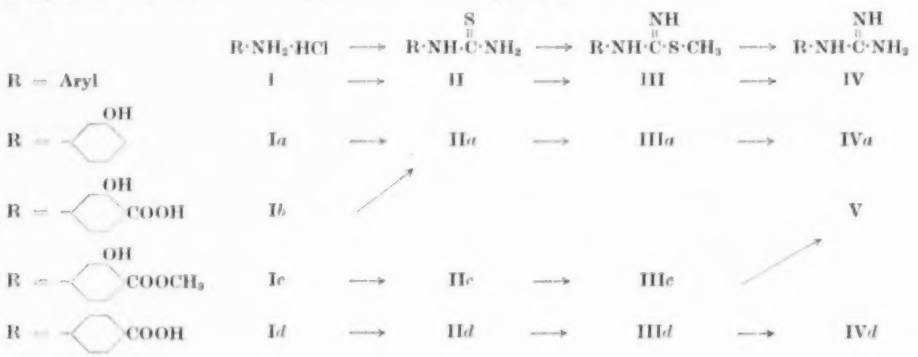
Six dyes, which directly dyed cotton brilliant green, were made from cyanuric chloride. C. O. C.

Fading of Azo Dyes. B. M. Bogoslovsky, Z. S. Kazakova, and M. M. Shemyakin. *J. Appl. Chem. U.S.S.R.*, **26**, 435–438 (April 1953).

Hydroxyazo dyes (nine naphthyl-, bromonaphthyl-, and nitronaphthyl-azonaphthols and the six *p*-(nitrophenylazo)- and *p*-(chlorophenylazo)-phenols) are dissolved in aq.–alcoholic NaOH and in aq. acetone, and the solutions are exposed to direct sunlight in presence and in absence of oxygen. Fading occurs only in presence of oxygen and is then very rapid for the azonaphthalene derivatives, but scarcely appreciable (after a six-months' exposure) for the azobenzene derivatives. Fading of the azonaphthalene derivatives is somewhat more rapid in the alkaline medium. A. E. S.

Synthesis of *p*-Guanidinosalicylic Acid and some Related Compounds. H. C. Beyerman and J. S. Bontekoe. *Rec. Trav. chim.*, **72**, 643–652 (July 1953).

The sequence in which the aryl-substituted thioureido compound resulting from the reaction of an arylamine hydrochloride with ammonium thiocyanate is methylated, and then converted into the corresponding arylguanidine by means of ammonia, has been developed according to the following scheme; the course of successful reactions is shown by arrows.



Compound V, 4-guanidinosalicylic acid, was synthesised also by (i) the reaction of *Ib* with cyanamide, and (ii) the saponification with ammonia of IV*c*, obtained by the reaction of I*c* with cyanamide. The ester (VI) resulting from the reaction of I*c* with dicyanimide, yields, on saponification with ammonia in pyridine, 4-*N'*-cyanoguanidinosalicylic acid (VII). Saponification of VI with alcoholic NaOH yields IV*a*.

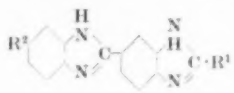
Aromatic amines sometimes yield *N*-aryl-*N'*-cyanoguanidines on diazotisation, coupling with guanidine, and decomp. of the diazonium group. The diazonium chloride of 4-aminosalicylic acid does not undergo these further reactions but couples "intermolecularly" to give 5:4'-dicarboxy-2:4:3'-trihydroxyazobenzene (VIII). Two other compounds—the dimethyl ester of VIII (IX) and the product of its etherification with diazomethane (X)—were prepared also. Full details are given of the preparation of II*a*, II*c*, II*d*, III*c*, III*d*, IV*a*, IV*c*, IV*d*, V, VI, VIII, IX, and X. J. W. D.

Absorption Spectra of the Direct Dye Benzopurpurin 4B and Related Structures. H. A. Standing and M. Stein. *J. Textile Inst.*, **44**, T 224–T 229 (May 1953).

The disazo compound obtained by coupling tetrazotised *o*-tolidine with naphthionic acid (Benzopurpurin 4B, referred to as *ortho*-Benzopurpurin) is widely used as a dye, whereas the isomeric compound from *m*-tolidine has no application. It is shown that the absorption spectrum of the *ortho* dye is very different from that of the corresponding half-structure (sodium *o*-tolylazonaphthionate), whereas that of the *meta* "dye" is similar to the corresponding half structure (sodium *m*-tolylazonaphthionate); the spectra of the half-structures are almost identical. The difference in spectrum between the disazo isomers is attributed to steric hindrance to planarity in the molecule of the *meta*-isomer, which has already been shown to be responsible for the lack of affinity for cellulose. J. W. B.

Glyoxaline Derivatives. VI—Synthesis of some Polybenziminazoles. B. A. Poray-Koshits, L. S. Efros, and E. S. Boychinova. *J. Gen. Chem. U.S.S.R.*, **23**, 835–841 (May 1953).

Benziminazole analogues of Primuline bases (benzothiazole derivatives) have not hitherto been obtained. Compounds of this type are now prepared by treating a benziminazole-5-carboxylic acid (prepared by oxidation of the 5-methyl compound) with an *o*-diamine (to give I), and by oxidising I ($R^2 = CH_3$) further to a carboxylic acid and condensing the product with an *o*-diamine (to give II). The absorption max. of benziminazole, I, and II occur at progressively higher wavelengths in the ultra violet (275, 315, and 345 mμ.), but at much shorter wavelengths than those of similar thiazole compounds.



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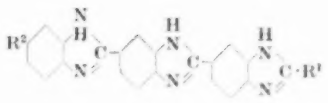


(II)

(R^1 = H or CH_3; R^2 = H or CH_3). A. E. S.

Glyoxaline Derivatives. VII—Preparation of Benziminazolesulphonic Acids by Baking. L. S. Efros. *J. Gen. Chem. U.S.S.R.*, **23**, 842–943 (May 1953).

Benziminazole- and 2-methylbenziminazole-5-sulphonic acids are prepared by baking the corresponding benziminazole sulphates (from 1 mol. of base and 1·2 mol. of H_2SO_4) at high temp. (up to 290°C.). A. E. S.

Glyoxaline Derivatives. VIII—Nitration and Azo-coupling Reactions of 4-Aminobenziminazole Derivatives. IX—Synthesis of 4-Aminobenziminazoles and a Comparison of their Chemical Properties with those of 5-Aminobenziminazoles. L. S. Efros. *J. Gen. Chem. U.S.S.R.*, **23**, 951–957, 957–963 (June 1953).

A number of reactions of 4-amino- and 4-amino-2-methyl-benziminazole, which may be prepared by the action of formic acid and acetic acid respectively on 1:2:3-triaminobenzene, are described. The 4-amino group greatly activates the benzene ring—coupling occurs with diazotised aniline with formation of reddish-orange *p*-aminoazo dyes, and the acylated compounds are readily nitrated in the *ortho* and *para* positions. When the amino group is in the 5-position, however, it has little effect on the activity of the benzene ring—thus, 5-aminobenziminazoles will not couple, even with very active diazo compounds—but potentiometric titration shows that it greatly increases the basicity of the glyoxaline ring, which is unaffected by a 4-amino group. A. E. S.

Synthesis of Leuco Bases and Dyes of the Triphenylmethane Series—III. I. Kh. Feldman and A. I. Zitser. *J. Gen. Chem. U.S.S.R.*, **23**, 676–680 (April 1953).

The nitro group of 4:4'-dihydroxy-3:3'-dimethoxy-3''-nitro-triphenylmethane (see J.S.D.C., **69**, 303 (Aug. 1953)) is reduced to amino by hydrogen in presence of Raney nickel and is converted further into acetamido, *p*-acetamidobenzenesulphonamido, and *p*-sulphanilamido. The substances so formed and the dyes obtained by their oxidation are described. A. E. S.

Influence of Addition of Potassium Polyvinyl Sulphate upon the Absorption Spectra and Fluorescence of Aqueous Rhodamine 6G. N. Mataga and M. Koizumi. *J. Inst. Polytech., Osaka City Univ.*, Ser. C, **3**, 21–35 (1952): *Chem. Abs.*, **47**, 8376 (25 Aug. 1953).

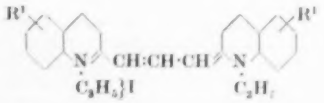
When metachromasy occurs, the added substance may affect (*a*) the energy levels within the dye by direct electrostatic action and/or (*b*) the state of aggregation of the dye molecules. The present case appears to be pure type (*b*). As the concentration of an aq. soln. of Rhodamine 6G is increased, the intensity of absorption at 526 mμ. decreases, while that at 500 mμ. increases; these two bands are accordingly ascribed to monomer and dimer respectively. When K polyvinyl sulphate is added to such a solution, the intensity of absorption due to dye monomer decreases while that due to dimer increases, without shift in position of the maximum; at higher concentrations of the sulphate the changes are reversed. It seems that, when the amount of added sulphate is small, adsorption causes increase in dye concentration on the surface of the particles and favours dimerisation; when the amount of sulphate is large, practically all the dye is adsorbed, and further addition of sulphate decreases the surface concentration of the dye. Assuming that monomer and dimer are adsorbed on the sulphate independently and proportionally to their concentrations in the solution, the following relation is derived—

$$\frac{y^2}{ZK} = \frac{(1 + a_M p)^2}{1 + a_D p}$$

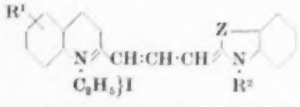
(M and D refer to monomer and dimer respectively; y = total concentration of monomer; Z = total concentration of dimer; K = equilibrium constant for dimerisation in the soln.; $a = ks/V$; k = ratio of concentration of dye adsorbed to concentration of dye in soln.; s = surface area of the sulphate per gram.; V = total volume; p = wt. of sulphate). This equation predicts that y^2/ZK should be independent of total dye concentration and, for large p, a linear function of p; the experimental data are in accord. Fluorescence intensity qualitatively parallels absorption intensity due to monomer, a relation which indicates that only the monomer fluoresces; there are quantitative differences ascribable to self-quenching by collision or to the effect of adsorption. Behaviour of other dyes is discussed on the basis of this theory. C. O. C.

Cyanine Dyes. I— Quino-, Indoquino-, and Thiaquino-carbocyanines. G. T. Pilyugin and Z. Ya. Krayner. *J. Gen. Chem U.S.S.R.*, **23**, 634–643 (April 1953).

The syntheses and properties are described of nine dyes of formula—



and twelve dyes of formula—

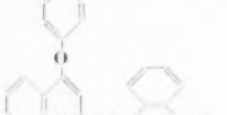


(R^1 = H or 5- or 6-NO_2, -NH_2, -NH·CO·CH_3, -NH·CO·C_6H_5 or -$N(CH_3)_2$; R^2 = CH_3 or C_2H_5; Z = S or $C(CH_3)_2$). In the symmetrical dyes, all substituents (R^1)— apart from 5-NH_2, which is without effect— produce bathochromic shifts, which are more pronounced in the 6-substituted compounds. The absorption max. of each unsymmetrical dye is compared with those of the two corresponding symmetrical dyes. The presence of an amino and an acetamido group results in an absorption max. that is displaced towards the shorter waves (by 2–13 mμ. when Z = S; 13–29 mμ. when Z = $C(CH_3)_2$) from the mean of the max. of the corresponding symmetrical dyes.

A. E. S.

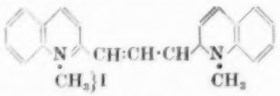
Cyclic Ammonium Dyes. III— Formation of Styryl and *iso*Cyanine Dyes from Quaternary Quinoline Derivatives. M. Katayanagi. *J. Pharm. Soc. Japan*, **68**, 133–135 (1948); **IV— Preparation of Styryl and Cyanine Dyes from Quaternary Quinaldine Salts.** *Ibid.*, 136–139: *Chem. Abs.*, **47**, 7503 (10 Aug. 1953).

III— Aromatic aldehydes and 4-phenoxyquinaldine methiodide yield 4-aminostyryl dyes with primary or secondary amines as catalyst but only a styryl compound if a tertiary amine is the catalyst. With purified amines (piperidine, triethylamine, and 1-methylpiperidine) and the following aldehydes— piperonal, opianic acid, and *p*-dimethylaminobenzaldehyde— it was found that (1) when very little amine was used or the amine was neutralised by acetic acid to bring the solution to pH 8–10·7, neither the 4-C_6H_5·O nor the 2-CH_3 group of the quinaldine showed any reactivity. (2) When the solvent contained >50% H_2O, only the 4-C_6H_5·O group was displaced, giving in the case of piperidine colourless crystals of 4-piperidinoquinaldine methiodide. When ethanol was the solvent the 2-CH_3 group was also activated, and a 4-amino-2-styryl dye was obtained. (3) When >0·5 mol. of a tertiary amine was used, 4-phenoxy-1:1′:2′-trimethyl-2:4′-quinocyanine iodide—



was formed, indicating that the 4-phenoxy-2-styryl dye primarily formed was decomposed and changed to the cyanine dye. (4) 2-*p*-Aminostyryl dyes formed by use of primary and secondary amines did not change to cyanine dyes, probably because *p*-amines do not readily decompose. (5) Making the solution more alkaline with KOH resulted in the hydrolysis of the 4-C_6H_5·O group to OH, giving 4-hydroxy*iso*cyanine dyes.

IV— Condensation of aldehydes and 4-phenoxyquinaldine methiodide proceeds in two directions according to the amount of amine used, one resulting in styryl- and the other in cyanine-type dyes. Piperonal and 4-phenoxyquinaldine methiodide in presence of trimethylamine yielded styryl dyes, the yield being 32·2% when $\frac{1}{10}$ mol. of amine was used, but decreasing with increase in amine content, finally becoming zero when 0·5 mol. of amine was used. Conversely, the yield of cyanine-type dyes was 2·45% when $\frac{1}{10}$ mol. of amine was used, but increased to a maximum of 37·6% with 0·5 mol. of amine. This indicates that the styryl dye formed is cleaved at the double bond of the vinyl group in presence of excess amine and changes to the more stable *iso*cyanine type. The fact is also explainable by the following reactions: 2-β-anilinovinylquinaldine methiodide is obtained from C_6H_5·NH·CH:N·C_6H_5 with α-piperonylidene-, α-vanillylidene-, or α-benzylidene-quinaldine methiodide, but when these anilino-type dyes are heated to ca. 160°c. with the latter three methiodides in presence of potassium acetate and acetic anhydride, cryptocyanine—

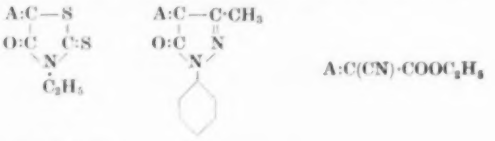


is formed. This is formed also when α-piperonylidene-quinaldine methiodide is heated to 160°c. with C_6H_5·NH·CH:N·C_6H_5, potassium acetate, and acetic anhydride.

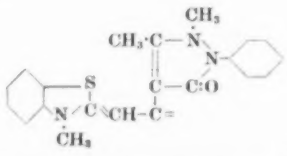
C. O. C.

Trinuclear Cyanine Photographic Sensitisers — III. A. van Dormael and J. Nys. *Bull. Soc. chim. Belg.*, **62**, 199–204 (March–April 1953).

New trinuclear *mero*cyanine dyes, which are photographic sensitisers, are obtained by reaction of a ketomethylene compound with a 2-(β-methylthio-β-4-antipyrinyl)vinyl*cyclo*ammonium salt; they are readily converted to the corresponding mono- and tri-methin rhodacyanines. Optical data are included and compared with those from the corresponding unsubstituted *mero*cyanines. The following trinuclear *mero*cyanines are described—



where A is —



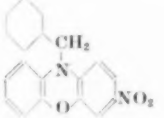
H. H. H.

Dyes of the Type of Aniline Black. J. Rais and J. Stamberg. *Chemie* (Prague), **8**, 30–31, 46–48 (1952): *Chem. Abs.*, **47**, 7781 (10 Aug. 1953).

A review with 48 references. C. O. C.

Aminophenoxazines. I— New Method for the Synthesis of 3-Aminophenoxazine. B. Boothroyd and E. R. Clark. *J.C.S.*, 1499–1504 (May 1953).

10-Benzyl-3-nitrophenoxazine (I) is synthesised, and when reduced by hydrogen in *n*-butyl alcohol with 10% palladium chloride–charcoal as catalyst gives 3-aminophenoxazine.



(I)

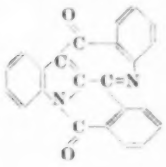
H. H. H.

Aminophenoxazines— II. B. Boothroyd and E. R. Clark. *J.C.S.*, 1504–1508 (May 1953).

A number of 1- and 3-aminophenoxazines are synthesised, including 3-amino-8-chloro-1-methyl-, 1-amino-8-chloro-, 3-amino-1-methyl, and 1:3-diamino-phenoxazine, and 3-amino-phenoxazine-1-carboxylic acid. H. H. H.

Constitution of Ciba Yellow 3G. R. S. Staunton and A. Topham. *J.C.S.*, 1889–1894 (June 1953).

It is shown that, of the five structural formulæ proposed for Ciba Yellow 3G, only I is consistent with the formation of the new degradation products II and III, whose structures are here established.



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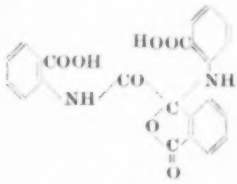
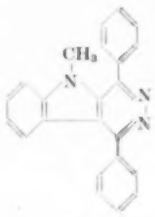


(II)



(III)

H. H. H.

Heterogeneous Redox Systems—Methylene Blue—Leuco Base of Methylene Blue. H. J. C. Tendeloo and T. de Vries. *Rec. Trav. chim.*, **72**, 377–389 (May 1953).

Potentiometric titrations are carried out using Mohr's salt–permanganate, iodine–thiosulphate, and Methylene Blue–titanous chloride (reoxidised with potassium dichromate) systems. All reactions are carried out with and without the addition of active carbon; it is shown that Methylene Blue when absorbed has markedly different properties from unadsorbed, and that its reduction by titanous chloride is hindered. J. W. B.

Spectra of Anthraquinone Derivatives. R. H. Peters and H. H. Sumner. *J.C.S.*, 2101–2110 (July 1953).

The absorption spectra of a number of mono- and di-substituted anthraquinones are recorded, and the effect of the substituents on the visible colour of anthraquinone is correlated with the electromeric possibilities of the groups. The ability of the substituents to participate in internal hydrogen bonds and the presence of steric interactions in the molecules affect the wavelength of maximum absorption. Interaction occurs between 1:4-substituents, but is less marked in the 1:5-compounds. Methylene groups, and both *m*- and *p*-phenylene residues are found to act as excellent chromophoric insulators in a series of acylated aminoanthraquinones and for certain derivatives containing two anthraquinone nuclei. H. H. H.

Reversible Appearance and Disappearance of Coloured Modifications of Some Compounds as a result of Irradiation at Low Temperatures. Y. Hirshberg, E. H. Frei, and E. Fischer. *J.C.S.*, 2184–2185 (July 1953).

When a solution of 1:3:3-trimethylindoline-2-*spiro*-6′-(2′:3′-*β*-naphthopyran) in ethanol–methanol mixture at 173° K. is irradiated in the 365 mμ. region, an intense mauve coloration is produced. At this temperature, both coloured and colourless modifications are stable. Irradiation with yellow light corresponding to the absorption maximum of the coloured modification in the visible region results in a steady decrease in colour intensity. The cycle of colour formation by ultraviolet, and its eradication by yellow irradiation, can be repeated many times. H. H. H.

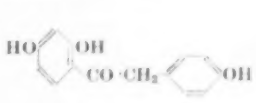
Polycyclic Systems. V—A New Route to Azulenes. E. A. Braude and W. F. Forbes. *J.C.S.*, 2208–2216 (Aug. 1953).

Chemistry of Fungi. XXI—Asperxanthone and a Preliminary Examination of Aspergillin. N. A. Lund, A. Robertson, and W. B. Whalley. *J.C.S.*, 2434–2439 (Aug. 1953).

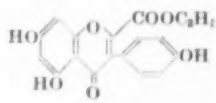
The black amorphous pigment aspergillin is isolated from several varieties of *Aspergillus niger*, and, after removal of iron and nitrogenous material, is oxidised by H_2O_2 to give small yields of acetaldehyde, oxalic acid, and mellitic acid, by $KMnO_4$ to give these two acids, and by HNO_3 to give all three compounds along with a little picric acid. A new 1-hydroxydimethoxymethylxanthone, asperxanthone $C_{12}H_5O_2(OCH_3)_2CH_3$, is isolated from the mycelium of several strains of *A. niger*, which on demethylation gives nor-rubrofusarin. H. H. H.

New Synthesis of *iso*Flavones—I. W. Baker, J. Chadderton, J. B. Harborne, and W. D. Ollis. *J.C.S.*, 1852–1860 (June 1953).

Benzyl *o*-hydroxyphenyl ketones (e.g. I) react at room temperature with ethoxalyl chloride in pyridine to give 2-carbethoxy*iso*flavones (e.g. II) in high yield. Hydrolysis to the carboxylic acids followed by thermal decarboxylation gives the *iso*flavones (e.g. III). The new synthesis is satisfactory with the tri- and poly-hydroxylated derivatives, and is particularly suited for the preparation of polyhydroxy- and partly alkoxylated *iso*flavones. Twelve *iso*flavones, six of them occurring naturally, are synthesised, viz. the 7-hydroxy, 7-hydroxy-3′:4′-methylenedioxy (*γ*-haptigenin), 7:4′-dihydroxy (daidzein), 4′-hydroxy-7-methoxy (formononetin), 5:7-dihydroxy, 5:7-dihydroxy-3′:4′-methylenedioxy, 5:7:4′-trihydroxy (genistein), 5:7-dihydroxy-4′-methoxy (biochanin-A), 5:4′-dihydroxy-7-methoxy (prunetin), 7:4′-dihydroxy-5-methoxy, 7-hydroxy-4′-nitro, and 5:7-dihydroxy-4′-nitro derivatives. The method, which may be used on a fairly large scale, involves direct *C*-ethoxalylation of the reactive methylene group and cyclisation to 2-carbethoxy-2-hydroxy*iso*flavones.



(I)



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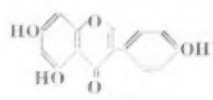
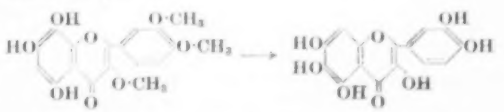


(III)

H. H. H.

Rearrangement of 5:8-Dihydroxyflavonols by Treatment with Hydriodic Acid. D. M. Donnelly, E. M. Philbin, and T. S. Wheeler. *Chem. and Ind.*, 567 (6 June 1953).

When 5:8-dihydroxyflavonols are treated with HI in phenol at 180–190° C. under pressure, molecular rearrangement takes place—



J. W. B.

New Synthesis of *iso*Flavones. II—5:7:2′-Trihydroxy-*iso*flavone. W. Baker, J. B. Harborne, and W. D. Ollis. *J.C.S.*, 1860–1864 (June 1953).

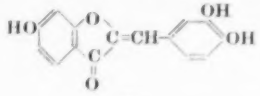
Isolation of 2′:4:4′-Trihydroxychalkone from Yellow Varieties of *Dahlia variabilis*. E. C. Bate-Smith and T. Swain. *J.C.S.*, 2185–2187 (July 1953).

2′:4:4′-Trihydroxychalkone has been chromatographically separated from butein in the hydrolysed extracts of the yellow varieties of *Dahlia variabilis*, by absorption on a column of Magnesol (a synthetic magnesium silicate), followed by elution with acetone, application to a column of Magnesol–Celite 545 (4 : 1) and development with dry acetone. 2′:4:4′-Trihydroxychalkone has not previously been isolated from natural sources, and its occurrence with

butein gives further weight to the view of a common precursor. H. H. H.

Isolation of a Benzylidenecoumaranone (Aurone) from a Yellow Dahlia. C. G. Nordström and T. Swain. *Chem. and Ind.*, 823–824 (1 Aug. 1953).

2:4:4′-Trihydroxychalkone has recently (cf. previous abstract) been chromatographically isolated from the hydrolysed petal extracts of Pius IX, a yellow variety of *Dahlia variabilis*, together with a second deep orange compound which was then thought to be butein, but which is now considered to be benzylidenecoumaranone (aurone) (I). Shimokoriyama and Hattori's sulphuretin from *Cosmos sulphureus*, to which the structure I is assigned, is either not identical with aurone or is a mixture.



(I)

H. H. H.

Plant Pigments. V— Glycosidic Structure of Astilbin. K. Hayashi and K. Ouchi. *Misc. Rept. Research Inst. Nat. Resources*, (26), 22–26 (1952): *Chem. Abs.*, **47**, 7493 (10 Aug. 1953).

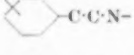
Astilbin is shown to be 3-rhamnosidyldistylin. C. O. C.

Iridescent Substance of Fish Scales— Pearl Essence. Y. Obata, H. Igarashi, and M. Ishida. *Bull. Japan. Soc. Sci. Fisheries*, **16**, 141–144 (1950): *Chem. Abs.*, **47**, 7682 (10 Aug. 1953).

Pearl essence from the scales of *Trichiurus haumela* was purified and shown to be guanine. Purified guanine is not iridescent. Long immersion of the essence in formalin destroyed the iridescence and treatment with pepsin increased the lustre; these facts suggest that the iridescence is due to an associated protein in the scales. Tyrosine, Zn stearate, BaS_2O_7, $PbHCO_3$, $KClO_3$, $MgNH_4PO_4$, and various Bi compounds, though having n_D >1·48, pearly lustre, and high m.p., and being freely crystalline and insoluble in organic solvents, are unsuitable as substitutes for pearl essence. C. O. C.

Oxidative Rupture of the Benzene Ring of 5:6-Dihydroxyindole and Congeners and the Biogenesis of Melanins. Z. E. Jolles. *Chem. and Ind.*, 845–846 (8 Aug. 1953).

Developments in the field of melanin biogenesis, since Angeli's theory of enzymatic oxidative fission was proposed in 1915, are summarised with 22 references. The recently reported ring opening between the OH groups of catechol and presumably at the corresponding positions of 5:6-dihydroxyindole (discovered by Raper in 1926 in the initial stages of the production of melanin from tyrosine by tyrosinase from the meal worm), simply by bubbling air through its alkaline solution, appears to justify a reconsideration of Angeli's theory. This is also based on results from biogenetic changes of molecules having in common the group—



H. H. H.

***In Vitro* Production of Melanin Pigment Granules.** J. L. Plummer and M. J. Kopac. *Pigment Cell Growth* (*Proc. 3rd Conf. Biol. Normal and Atyp. Pigment Cell Growth*), 305–317 (1953): *Chem. Abs.*, **47**, 8130 (25 Aug. 1953).

In presence of tyrosinase and either tyrosine or dopa, Dry flow starch grains (a hydrophobic starch preparation) became coated with a thin layer of melamin. Dry flow grains in presence of tyrosinase and *p*-cresol acquired a yellow coating. Addition of tyrosine to systems containing tyrosinase and dispersed substitutes not only showed a high rate of oxidation but also formed particles of melanin. Along with formation of pigment there was always appreciable oxygen consumption. Pigment formation requires first the oxidation of a susceptible substrate. Transformation of pigment particles into granules requires, in addition, a physical substrate, on which polymerisation and deposition of the oxidised phenolic substances may take place. C. O. C.

Structure of Melanin. H. S. Mason. *Pigment Cell Growth* (*Proc. 3rd Conf. Biol. Normal and Atyp. Pigment Cell Growth*), 277–303 (1953); *Chem. Abs.*, **47**, 8130 (25 Aug. 1953).

Review. C. O. C.

Origin and Development of Titanium Dioxide Pigments. S. A. Ray. *Verfkroniek*, **26**, 46–51 (1951): *Chem. Abs.*, **47**, 8388 (25 Aug. 1953).

A discussion of the occurrence, use as raw material, crystalline modifications, and physical constants of anatase and rutile; the chemical analysis of ilmenite; behaviour of TiO_2–ZnO mixtures; and the effect of pigment : binder ratio on reduction in gloss. C. O. C.

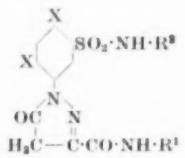
Lead Titanate. F. H. W. Wachholtz. *Verfkroniek*, **26**, 52–56 (1953): *Chem. Abs.*, **47**, 8387 (25 Aug. 1953).

Discussion of an ideal crystal structure of $PbTiO_3$ at high temperature, the measurements of an X-ray picture of a unit cell in dependence upon temperature, specific heat as a function of temperature, and change in gloss against time of exposure comparing $PbTiO_3$ with TiO_2 (the former being considerably the superior) and the weather resistance of coatings containing $PbTiO_3$ compared with other pigments. C. O. C.

PATENTS

Red Metachrome Monazo Pyrazolone Dyes. Gy. *BP* 697,517

Pyrazolone diamides—



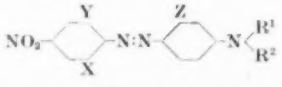
(R^1 and R^2 = H, Alk, or hydroxyalkyl; one X = Cl or H and the other X = H) are coupled with diazotised 4-alkyl- (or *cyclo*alkyl)-2-amino-6-nitrophenols to yield bluish-red metachrome dyes. Thus aniline-*m*-sulphonamide is converted into the corresponding hydrazine, which is condensed with sodium ethyl oxalacetate to produce ethyl 1-*m*-sulphamylphenylpyrazolone-3-carboxylate. Treatment with aq. NH_3 for 2–4 days at 20° C. or for 10–15 hr. at 80–100° C. produces the diamide (Xs = H; R^1 and R^2 = H). This is coupled with diazotised 2-amino-4-*tert*-amyl-6-nitrophenol to give the bluish red metachrome dye, which exhausts well from a neutral or weakly acid bath. E. S.

Monoazo Dyes from 1-Alkyl-3-methyl-5-pyrazolones. Ciba. *BP* 697,056

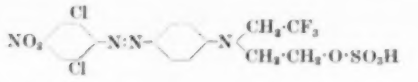
1-Alkyl-3-methyl-5-pyrazoles (Alk of >3 C) are made by treating the corresponding alkyl halide with hydrazine and condensing the resulting alkylhydrazine with acetoacetic ester or amide. By coupling with appropriate diazo compounds, pigments, disperse dyes, acid dyes, and afterchrome or metachrome dyes are prepared. Thus 2-aminophenol-5-sulphonamide → 1-*n*-butyl-3-methyl-5-pyrazolone is a reddish-orange metachrome dye. E. S.

Monoazo Dyes, containing Trifluoromethyl Groups, for Cellulose Acetate. Eastman Kodak Co. *USP* 2,618,630

Azo dyes of formula—



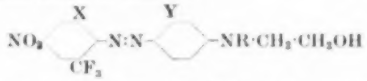
(R^1 = β-hydroxyethyl, γ-hydroxypropyl, or $\beta\gamma$-dihydroxypropyl; R^2 = $\beta\beta\beta$-trifluoroethyl or $\gamma\gamma\gamma$-trifluoropropyl; X = Cl or Br; Y = F, Cl, Br, CH_3, or CF_3, and Y also may = $CH_2{\cdot}SO_2$ when X = H; Z = H, Cl, Br, CH_3, or C_2H_5) dye acetate rayon orange, brown, red, and rubine. The corresponding sulphate compounds are also claimed. Thus the monoazo compound 2:6-dichloro-4-nitroaniline → *N*-$\beta\beta\beta$-trifluoroethyl-*N*-β-hydroxyethylaniline is dissolved in pyridine and treated with chlorosulphonic acid to yield the sulphato compound—



which dyes acetate rayon orange.

USP 2,618,631

Azo compounds of formula—



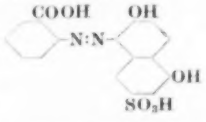
(X and Y = Cl or Br; R = CH_3, C_2H_5, $HO{\cdot}CH_2{\cdot}CH_2$, $HO{\cdot}CH_2{\cdot}CH_2{\cdot}CH_2$, or $CH_3{\cdot}CH(OH){\cdot}CH_2$) are brownish-orange disperse dyes for cellulose acetate. Thus, 2-chloro-4-nitro-6-trifluoromethylaniline is diazotised by means of nitrosylsulphuric acid and coupled with *NN*-bis-β-hydroxyethyl-*m*-chloroaniline. E. S.

Violet Azoic Dyes. Ciba. *BP* 697,480

By using as a diazo component a 4-benzoyl(or phenoxyacetyl)amino-2:5-dimethoxyaniline in which the benzene nucleus of the acyl group may contain substituents not imparting solubility in water, and as a coupling component the *m*-2-xylidide of 3-hydroxy-2-naphthoic acid, violet azoic dyeings and prints are obtained. Thus cotton yarn is impregnated with a solution of the *m*-2-xylidide of 3-hydroxy-2-naphthoic acid in aq. NaOH, and then worked in a bath containing diazotised 4-benzamido-2:5-dimethoxyaniline to develop the bluish-violet azoic dye. E. S.

o-Hydroxyazo Dyes. S. *BP* 697,488

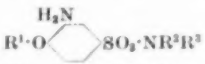
The *o*-aminoazo compounds obtained by acid-coupling J acid, or 1-amino-7-naphthol-3(or 4)-sulphonic acid, with diazo compounds are converted into *o*-hydroxyazo compounds by treatment with nitrous acid or other mineral acid or with caustic alkalis. The products may be coupled further with appropriate diazo or diazoazo compounds to give metallisable dis- and poly-azo dyes. Thus the monoazo dye obtained from the acid coupling of diazotised anthranilic acid with J acid is heated in an autoclave at 140–150°C. for 8 hr. with 2·5% aq. NaOH to yield the *o*-hydroxyazo compound



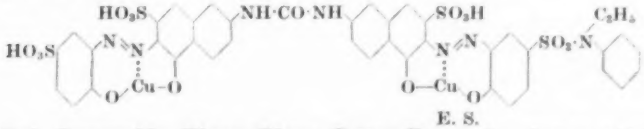
E. S.

Metal (Copper)-complex Disazo Direct Cotton Dyes. S. *BP* 695,748

Urea-di-J acid is coupled with 1 mol. of diazotised 2-aminophenol-4-sulphonic acid (or its methyl ether) and with 1 mol. of a diazotised 2-aminophenol-4-sulphonamide—



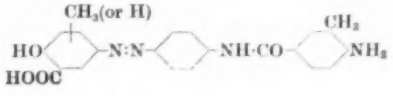
(R^1 = H or CH_3; R^2 = H or Alk; R^3 = H, Alk, or subst· or unsubst. phenyl), and the resulting disazo compounds are converted into copper complexes, which dye cellulose bright bordeaux. Thus diazotised 2-aminophenol-4-sulphon-*N*-ethylanilide is coupled with urea-di-J acid in presence of $NaHCO_3$; soda ash is then added, and coupling carried out with diazotised 2-aminoanisole-4-sulphonic acid. The copper complex of the resulting disazo compound has the formula—



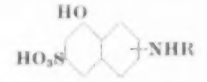
E. S.

Red, Copperable, Disazo Direct Cotton Dyes. S. *BP* 695,758

Aminoazo compounds—



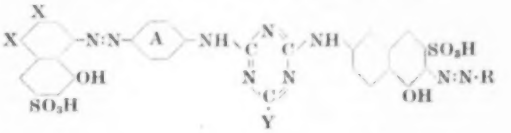
are diazotised and coupled with aminonaphtholsulphonic acids—



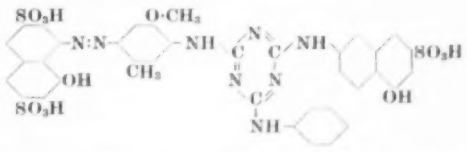
(R = H or acyl, and a further SO_3H may be present), the resulting dye being treated with acylating agents if R = H, to produce scarlet and red direct cotton dyes, which may be aftercoppered. Alternatively, if R contains a diazotisable NH_2 group, the dye may be diazotised on the fibre and developed with β-naphthol. Thus the monoazo compound *p*-nitroaniline→salicylic acid is reduced to the amino compound with Na_2S, acylated with 3-methyl-4-nitrobenzoyl chloride, and again treated with Na_2S to give the desired aminoazo compound. This is diazotised, and coupled with 2-carbethoxyamino-5-naphthol-1:7-disulphonic acid in presence of soda ash, to give a scarlet-red direct cotton dye, which becomes fast to washing and light on aftercoppering. E. S.

Metal (Copper)-complex Disazo Direct Cotton Dyes. Ciba. *BP* 697,416

The copper complexes of the disazo compounds—



(one X = H, the other X = SO_3H; Y = Hal, OH, NH_2, or NH·Ar; R = a benzene radical containing, *ortho* to the azo link, Hal, O·Alk, or OH, and preferably also $SO_2{\cdot}NH_2$ or SO_3H; A may contain substituents) are bordeaux direct cotton dyes, whose good light fastness is not reduced by application of a urea–formaldehyde crease-resist finish. Thus cyanuric chloride is condensed with (*a*) 1 mol. of J acid, (*b*) 1 mol. of the aminoazo dye *p*-toluenesulphonic ester of H acid→*p*-cresidine, and (*c*) 1 mol. of aniline. Removal of the *p*-toluenesulphonyl group by hydrolysis gives the product—



which is coupled with diazotised 2-aminoanisole-4-sulphonic acid. Heating with ammoniacal copper sulphate gives the required copper complex. E. S.

Dis- and Poly-azo Direct Cotton Dyes. Ciba. *BP* 697,136

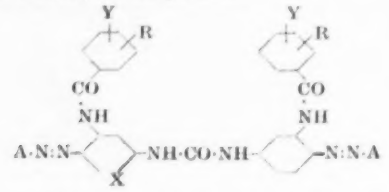
The aminoazo compound—

(the quinoline radical may contain e.g. a sulpho group) is diazotised and coupled (*a*) in alkaline medium with a 1-naphthol-3-sulphonic acid containing a substituted amino group in the 5 or 6 position; (*b*) in acid medium with J acid, the resulting disazo dye being alkali-coupled with a further diazo compound; or (*c*) in alkaline medium with a compound capable of coupling twice and containing two residues of J or γ acid linked together through their NH_2 groups, using either 2 mol. of the above diazotised aminoazo compound or 1 mol. of it and 1 mol. of another diazo compound. The resulting dis- and poly-azo compounds are direct cotton dyes, those derived from J acid or its substitution products being suitable for aftercoppering. Thus the monoazo compound 5-amino-2-nitro-benzoic acid→8-hydroxyquinoline is reduced at 45–50°C. with

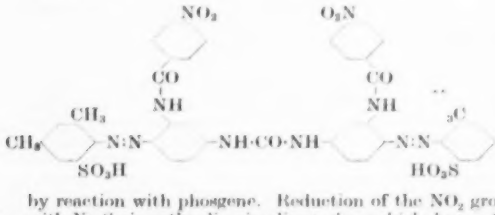
Na_2S to the required aminoazo compound, which is diazotised and coupled in presence of Na_2CO_3 with *N*-phenyl-J acid. The disazo dye so formed dyed cellulosic fibres violet, converted to reddish blue by treatment with a copper salt. E. S.

Polyazo Direct Cotton Dyes for Diazotisation and Development on the Fibre. S. *BP* 695,760

Dis- and poly-azo compounds—



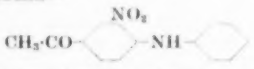
(A = aryl which contains at least one SO_3H, and may contain Hal, Alk, O·Alk, CO·NH·Ar, or N:N·Ar; X = H, Alk, or O·Alk; Y = H, Hal, Alk, or O·Alk; R = NO_2 or NH·Acyl) are converted into dyes, which may be diazotised and developed on the fibre, by converting the group R into NH_2 by reduction or hydrolysis. Thus the monoazo compound *m*-4-xylidine-5-sulphonic acid → *N*-4-nitrobenzoyl-*m*-phenylenediamine is converted into the urea derivative—



by reaction with phosgene. Reduction of the NO_2 groups with Na_2S gives the diaminodisazo dye, which dyes cotton a dischargeable yellow. Diazotisation on the fibre and development with phenylmethylpyrazolone improves the washing fastness. E. S.

Alkylketodiphenylamines—Disperse Dyes. General Aniline. *BP* 696,989

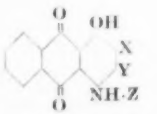
Fast-to-light yellow dyes for acetate rayon and nylon are prepared by condensing a 4-bromo(or chloro)-3-nitro-1-alkylketobenzene with a substituted or unsubstituted aniline. Thus 4-chloro-3-nitroacetophenone and aniline are refluxed with aq. $NaHCO_3$ for 16 hr., and the resulting compound—



is recrystallised from *iso*propyl alcohol. R. K. F.

Nuclear-monohalogenated 4-β-Fluoroalkylamino-1-hydroxyanthraquinones—Disperse Dyes. Eastman Kodak Co. *USP* 2,624,746

Compounds of formula—



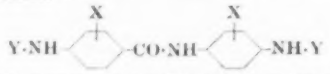
(X = Br or Cl when Y = H and *vice versa*; Z = 2:2-difluoroethyl, *n*-propyl, *-iso*propyl, or *-n*-butyl, or 2:2:2-trifluoroethyl or *-iso*propyl) are red to pinkish-orange dyes for cellulose aliphatic carboxylates containing 2–4 C in the acid group. They have outstanding fastness to light and gas-fume fading. Those having Br or Cl in the 2-position are made by brominating or chlorinating the corresponding 4-β-fluoroalkylamine-1-hydroxyanthraquinone. Those with Br or Cl in the 3-position are made by reaction of a 2:4-dibromo(or dichloro)-1-β-fluoroalkylaminoanthraquinone with boric acid and steam. C. O. C.

Arylaminoacedianthrone Dyes. ICI. *BP* 696,679

Acedianthrones carrying at least one arylamino group are converted to soluble *iso*thiouronium derivatives by being treated first with bischloro(or bromo)methyl ether and then with a thiourea to give brown dyes of good fastness to light on cellulosic fibres. Thus *p*-toluidinoacedianthrone (obtained e.g. by condensing nitroacedianthrone with *p*-toluidine) is stirred for 18 hr. at 15–20°C. in 96% H_2SO_4 with bischloromethyl ether, and then poured into ice and water. The product is heated at 85–90°C. for 30 min. with aq. tetramethylthiourea, and the resulting soluble dye separated by adding acetone at 60°C. and cooling to 25°C. R. K. F.

4:4′-Diaminostilbene Derivatives—Fluorescent Brightening Agents. FBy. *BP* 695,609

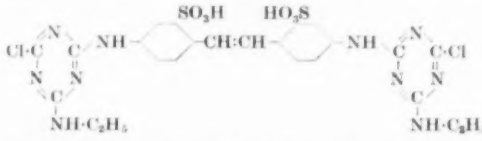
Wash-fast fluorescent brightening agents have the general formula—



(X = solubilising group, e.g. SO_3H; Y = univalent radical of the reaction product of 1 mol. of 4:4′-diaminostilbene-disulphonic acid and 2 mol. of cyanuric chloride, in which the Cl atoms may be replaced by e.g. NH_2 or OH). They are made by various methods. Thus an acetone solution of cyanuric chloride is poured into aq. sodium 4:4′-diaminostilbene-2:2′-disulphonate containing ice, while aq. Na_2CO_3 is added in amounts which absorb the acid formed by the reaction while leaving the solution acid to Congo red paper. Aniline is next added at 25–30°C., followed by aq. NaOH until neutral, and then an aq. solution of sodium 2-*p*-aminobenzamido-5-acetamidobenzenesulphonate. After heating at 60°C., aq. NH_3 is run in, the acetone distilled off, and the product precipitated by adding NaCl. R. K. F.

4:4′-Diaminostilbene-2:2′-disulphonic Acid Derivatives—Fluorescent Brightening Agents. Ciba. *BP* 696,296

Fluorescent brightening agents having affinity for cellulose and animal fibres are prepared by condensing cyanuric chloride (2 mol.) with 4:4′-diaminostilbene-2:2′-disulphonic acid (1 mol.), and then with an alkylamine-dialkylamine, *N*:*N*-dialkylalkylenediamine, or hetero, cyclic amine (2 mol.). Thus the compound—



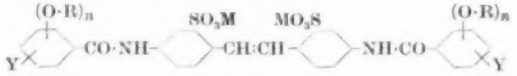
is made by adding aq. ethylamine at 10°C. to a neutral aq. soln. of the condensation product of cyanuric chloride and 4:4′-diaminostilbene-2:2′-disulphonic acid.

BP 696,357

Similar products are made by related methods to yield compounds in which one of the two remaining chlorine atoms is replaced by a substituted amino group and which may contain hydroxyalkylamino groups. R. K. F.

Fluorescent Brightening Agents. General Aniline. *USP* 2,623,064

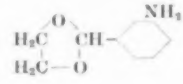
Fluorescent brightening agents of formula—



(M = H or other cation; R = unsat. short-chain aliphatic radical or a halogeno-subst. unsat. aliphatic radical of chain length preferably < 5 C; *n* = 1–3; Y = H or lower Alk or Alk·O) are prepared by condensing 1 mol. of 4:4′-diaminostilbene-2:2′-disulphonic acid with >2 mol. of an unsat. aliphatic ether of a hydroxybenzoyl chloride. C. O. C.

***m*-Dioxolanylphenylaminoanthraquinones—Acid and Disperse Dyes.** General Aniline. *USP* 2,612,507

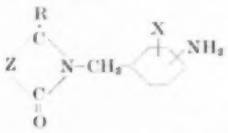
2-*m*-Aminophenyldioxolan—



is condensed in aq. soln. with 1-amino-4-bromoanthraquinone-2-sulphonic acid in the presence of Na_2CO_3, $NaHCO_3$, and Cu_2Cl_2 to yield a blue acid dye, which on further treatment with aq. NH_3 and $Na_2S_2O_4$ at 70–75°C. loses its SO_3Na to give a blue disperse dye having good resistance to burnt gas fumes. R. K. F.

Anthraquinonoid Wool Dyes containing a Lactam Ring. General Aniline. *USP* 2,615,021

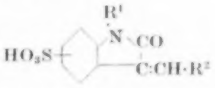
Aminobenzyllactams of formula—



(R = H or lower Alk; X = H, Hal, OH, Alk, Alk·O, COOH, or SO_2H; Z = alkylene necessary to complete a lactam ring) are condensed with 1-amino-4-bromoanthraquinone-2-sulphonic acid in aq. soln. containing Na_2CO_3, $NaHCO_3$, and Cu_2Cl_2, to produce green–blue wool dyes. The lactam derivative is prepared by treating a substituted nitrobenzene, e.g. *m*-chloronitrobenzene, with a lactam, e.g. γ-butyrolactam, in presence of formaldehyde, and reducing. R. K. F.

3-Benzylideneoxindolesulphonic Acids—Dyes. General Aniline. *USP* 2,622,980

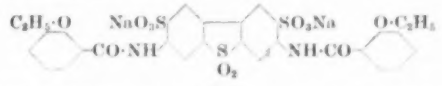
Dyes of formula—



(R^1 = H, Alk of <23 C, Ar, alkoxyphenyl, or aralkyl; R^2 = Ar of the benzene series which may be substituted by one or more OH, SO_3H, Alk, O-Alk, dialkyamino, pyridyl, or other monocyclic heterocyclic radical) are useful filter and antihalation dyes. They are obtained by condensing sulphonated oxindoles with benzaldehydes or aldehydes of furan, thiophen, and pyrrole. C. O. C.

Bisacylaminodibenzothiophen 5:5-Dioxide Derivatives—Fluorescent Brightening Agents. American Cyanamid Co. *BP* 696,038

The sulphonated 3:7-bisacylaminodibenzothiophen 5:5-dioxides of *BP* 684,842 (*cf.* J.S.D.C., **69**, 95 (March 1953)) are prepared by treating a salt of the non-acylated sulphonic acid and a tertiary amine (or other organic base) with an acylating agent in a non-polar solvent of b.p. >75°C. Thus 3:7-diamino-2:8-disulphodibenzothiophen dioxide is treated with triethylamine in dil. H_2SO_4 soln. The resulting salt is slurried in chlorobenzene and refluxed after addition of triethylamine and *o*-ethoxybenzoyl chloride. The product after steam-distillation and precipitation with NaCl is—



It is fast to chlorine and does not discolour a detergent powder of which it is part. R. K. F.

Phthaloyldibenzothiophen Derivatives—Dyes for Acetate Rayon and Vat Dyes. General Aniline. *USP* 2,615,898

α-Amino groups are introduced into the phthaloyldibenzothiophens of *USP* 2,533,171 (J.S.D.C., **67**, 467 (1951)) to produce a range of dyes (orange to blue) for acetate rayon. Treatment with acylating agents converts these dyes into vat dyes. Thus the condensation product of dibenzothiophen and phthalic anhydride is dissolved in H_2SO_4 and nitrated by adding an HNO_3–H_2SO_4 mixture at 40°C. After separating by pouring into water, the resulting α,α'-dinitro compound is reduced with aq. Na_2S to give a scarlet disperse dye. R. K. F.

Benzophenothiazine Dyes. American Cyanamid Co. *USP* 2,607,774

Dyes having differential tissue-staining properties are prepared by condensing a 2-amino-5-dialkylaminobenzenethiosulphuric acid with an *N*-aryl-1-naphthylamine. Thus the dye—

is made by adding aq. $K_2Cr_2O_7$ to a soln. of 2-amino-5-diethylaminobenzenethiosulphuric acid and *N*-phenyl-1-naphthylamine in glacial acetic acid containing a little HCl at room temperature. After warming to ca. 100°C. for 2 hr. and pouring into water, the resulting product is dried and the dye leached out with boiling benzene. R. K. F.

Non-crystallising Copper Phthalocyanine. General Aniline. *USP* 2,615,026

Copper phthalocyanine is heated at 185–187°C. in a melt of $AlCl_3$ and NaCl. The product after acid pasting does not crystallise when in contact with organic solvents. *USP* 2,615,027

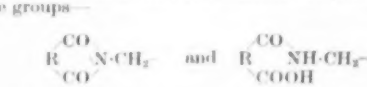
A copper phthalocyanine of similar properties is prepared by having present during its synthesis from phthalic anhydride, urea, Cu_2Cl_2, and a catalyst in trichlorobenzene, 5·7·5% $AlCl_3$ calculated on the wt. of phthalic anhydride. R. K. F.

Phthalocyanine Vat Dyes. DuP. *USP* 2,613,129

Cobalt phthalocyanine is treated with PCl_3 in a melt of NaCl and $AlCl_3$ at 120–130°C. and poured into water, when the entering PCl_2 groups are hydrolysed to $P(OH)_2$ groups. The product, a strong blue vat dye, contains 1·35% P which corresponds to about 3 $P(OH)_2$ groups per 10 phthalocyanine mol. R. K. F.

Imidomethylated Phthalocyanines. American Cyanamid Co. *BP* 695,523

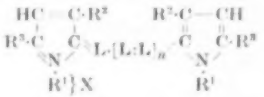
Phthalocyanine derivatives substituted by one or more of the groups—



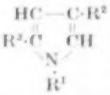
(R = 1:2-bivalent radical) show resistance to crystallising in organic liquids and are readily converted to aminomethylphthalocyanines, which are useful as dye intermediates. They are made by treating a phthalocyanine with a hydroxymethylimide or with reagents which produce a hydroxymethylimide under acidic anhydrous conditions. Thus copper phthalocyanine is heated at 50–55°C. in 95% H_2SO_4 with paraformaldehyde and phthalimide, and finally poured into ice and water. R. K. F.

Methin Cyanine Dyes. Kodak. *BP* 696,646

Dyes of formula—



(R^1 = Alk of 1–18 C, aralkyl, or *cyclo*alkyl; R^2 and R^3 = subst. or unsubst. Ar; L = subst. or unsubst. methin; *n* = 0–2; X = anion) are obtained by condensing a compound of formula—



with formic acid, NCHO, a diarylformamidine, a trialkyl orthocarboxylate, "benzotrichloride" (benzylidene chloride), a monocarboxylic acid halide or benzoyl chloride. They are useful in photographic filters. C. O. C.

Caramel. Union Starch & Refining Co. *BP* 696,736.

Caramel colour resistant to acids and other coagulants and to thickening on ageing is produced by heating a concentrated solution of the raw carbohydrate with acid, neutralising (preferably with anhydrous ammonia), heating to 230–270°F. for 15–120 min., cooling to 190–230°F., and then adding the final reagents and caramelising. A brilliantly clear product is obtained. C. O. C.

Furnace Black. J. M. Huber Corpn. *USP* 2,623,811
A method of making furnace black which yields valuable by-product gases.

USP 2,625,466
A very finely divided black is obtained in good yield if the hydrocarbon is rapidly decomposed while highly diluted. The length of time that the carbon is kept hot is not so important, and does not seem to be critical.
C. O. C.

Zirconium–Vanadium–Titanium Oxides—Ceramic Pigment. Harshaw Chemical Co. *USP* 2,623,833
Calcination of ZO_2, containing 0·2–2·0% by wt. of Na_2O and <5% of free SiO_2, with V_2O_5 or NH_4VO_3 and TiO_2 yields a brown product, which when fired in glazes or bodies yield an intense yellow, slightly browner than lemon yellow.
C. O. C.

Fundamental Properties of Dyes (VIII p. 470).
Substituted Benzidines and Related Compounds as Reagents in Analytical Chemistry. XII— Reagents for the Precipitation of Sulphate (XIV p. 478).
Analysis of Flavones. I— Stoichiometry of Dissolved Metal-salt Complexes of Rutin and Quercetin (XIV p. 478).

V— PAINTS; ENAMELS; INKS

PATENTS

Zein Inks. Time. *USP* 2,622,988
Inks which set almost instantaneously at 80–90°F. and R.H. <75%, but which are stable at temperatures near the dew point of the atmosphere in which they are used, are based on zein dissolved in a mixture of water and a non-solvent for zein. The non-solvent used must form an azeotropic mixture with water, but the ratio of water to non-solvent must be less than in the azeotropic mixture.
C. O. C.

Ink for Ball-point Pens. Eversharp. *USP* 2,623,827
An ink which is normally solid, but is converted locally to a fluid by frictional or shearing contact with the rear surface of the writing ball, consists of a polyethylene or polyhydroxyethylene glycol of mol. wt. 400–16,000 dispersed in an acid salt of a basic dye, e.g. Victoria Blue oleate. Preferably the emulsion contains, in a third phase, a pigment which gives greater light fastness and permanency and helps to control the stability of the emulsions e.g. Prussian Blue or Monastral Blue.
C. O. C.

Controlling the Viscosity of Paints, etc. by Adding Modified Clays. National Lead Co. *USP* 2,622,987
The viscosity and stability of a paint can be increased by adding a modified clay, e.g. bentonite, which forms a gel in the organic vehicle used.
C. O. C.

Origin and Development of Titanium Dioxide Pigments (IV p. 462).
Lead Titanate (IV p. 462).

VI— FIBRES; YARNS; FABRICS

Review of Textile Research and Development during 1952. R. Steele, C. Messler, and J. H. Wakelin. *Text. Research J.*, **23**, 313–372 (May 1953).
In a bibliography are given 1997 references covering the main headings of protein fibres, cellulose fibres, synthetic fibres and other high polymers, molecular-weight determination, test methods and apparatus, fibre–yarn–fabric relationships, textile manufacturing, and dyeing and finishing.
A. B.

Ancient and Modern Textile Production in London and the Home Counties. E. Goodale. J.S.D.C., **69**, 317–328 (Sept. 1953).

Fibre Architecture with special reference to Molecular Constitution. S. J. Allen. *J. Textile Inst.*, **44**, P 286–P 306 (Aug. 1953).
Fibre architecture is outlined from the point of view of the polymer chemist, with emphasis on the explanation of such properties as draping quality, handle, and resistance to wear. The possibility of forming regenerated polymers like wool and silk is discussed.
J. W. B.

Investigation of the Frictional Properties of Textile Fibres under Variable Fibre Stress. B. G. Hood. *Text. Research J.*, **23**, 495–505 (July 1953).
An apparatus is described for the measurement of the relative frictional properties of individual textile fibres. Results are given for cotton, nylon, fortisan, ramie, viscose rayons, wool, and mohair.
A. B.

Mechanism of the Formation of Electrostatic Charges on Fibres. H. Grüner. *Faserforsch. und Textiltech.*, **4**, 249–260 (June), 275–287 (July 1953).
The problem of the sources of electrostatic charges on textiles is examined and relevant literature is reviewed (46 references). It is considered that the most probable sources are friction and contact effects (mainly) and deformation and heating of the fibre (to a minor extent). In an investigation of some effects leading to charge formation, in which an electrometer fitted with a stretched silvered Perlon thread is used, it is first established that a charge may arise from friction between two like surfaces—for all materials investigated (Perlon L, Orlon, Terylene, wool, silk, polystyrene, ebonite, viscose rayon, staple, glass, and amber), the rubbing surface having the greater area takes up a positive charge, and the smaller surface becomes negative. A number of possible explanations for this behaviour are considered. The difficulties arising in the quantitative investigation of the effect of friction between unlike surfaces are examined, and, in view of these difficulties, no investigation is attempted. Pure contact effects, however, are examined by passing one of the materials, in the form of yarn, through a nip formed by rollers contructed of or covered with the other material, the yarn being then passed into a Faraday pail attached to the electrometer. The effect of roller speed is found to be very small, but increase in nip pressure, and therefore of area of contact, leads to increased charge. On the basis of the results, the materials examined are placed in a potential series, from which the contact potential for any pair of the materials can be estimated. Thus Orlon, intermediate in the series between rubber and paper (in arbitrary units, +15 against rubber and −13 against paper), is found not to acquire any charge when passed through a rubber–paper nip; for the same reason, nylon 66 remains practically uncharged when passed through a brass–paper nip. The effect of an antistatic preparation (nature unknown) on the contact potential is small, its effectiveness in practice being mainly attributable to the increased conductivity that it imparts. It is further found that, when Perlon L filaments are broken, the two ends so formed generally take up opposite charges, the end of the portion that issued first from the spinneret during manufacture being charged positively in the majority of cases. This effect is considered to be associated with the "unidirectional" nature of the chain (the polar grouping NH·CO is constant in its orientation along the length of the chain; cf. nylon 66, in which NH·CO alternates with CO·NH) and with a preferred direction of orientation of the chain molecule, produced in the spinning process. Difficulties are met in the verification of this theory for other fibres, owing to the non-availability of these in suitable monofil form, and it is uncertain to what extent effects found for yarns are to be attributed to fibre friction. However, only very small effects are found for nylon 66 and Orlon yarns, and Terylene and viscose rayon yarns show no effect. It is found also, in accord with the above results, that a definite piezoelectric effect can be observed in a stretched Perlon fibre. The massive polymer is not piezoelectric, but if it is melted to form a thin layer and resolidified while subjected to a vertical electric field, the product gives a powerful piezoelectric effect (~20% of that of quartz) and also a strong pyroelectric effect (when the sheet is heated and then allowed to cool, a p.d. develops between its two faces).
A. E. S.

Mechanical Properties of Cotton Fibres. II— Effect of Acids on Fibres under Tension. J. A. Lasater, E. L. Nimer, and H. Eyring. *Text. Research J.*, **23**, 481–488 (July 1953).
The effect of acids on cotton fibres held under tension is investigated using relaxation constants before and after treatment. Hydrochloric, sulphuric, and nitric acids are used, and the data treated quantitatively in the case of hydrochloric and sulphuric acids.
A. B.

Heat of Wetting and Hydrophilic Character of Cellulose. A. V. Dumansky and E. F. Nekryach. *Kolloid. Zhur.*, **15**, 91–98 (1953): *Chem. Abs.*, **47**, 7880 (25 Aug. 1953).

The heat of wetting Q of cellulose dried *in vacuo* at 55°c. is 0·7–3·0 g.cal./g. greater than Q of cellulose dried in air at 105–120°c. When duration of drying at 55°c. is lengthened to 24 hr., Q increases, but is not effected by longer drying. The greatest values of Q are 13·8–15·0 for sulphite cellulose, 16·0–18·7 for sulphate cellulose, and 18·2 g.cal./g. for cellulose nitrate. Unexpectedly, there is no correlation between Q and the reactivity of cellulose toward CS_2. The heat of wetting by ethyl alcohol is small, ranging between −3·2 and +4·5 g.cal./g. The heat of wetting by aq. ethyl alcohol, as a function of the wt. % of water, gives an S-shaped curve. These observations are similar to those on starch, gelatin, and agar. Q of cellulose by water is greatly lowered by slight moistening (e.g. halved by 1% water); this behaviour is peculiar to cellulose. The water content A at which $Q = 0$ (by extrapolation) agrees with the equation $A = Q/80$. Q of wetting by ethyl alcohol first becomes more negative and then positive as the A of cellulose is increased. 1–2 mol. of water are attached to each glucose residue. C. O. C.

Further Observations on the Effects of Evaporating Water from Cotton Cellulose. G. W. Madaras and H. A. Turner. J.S.D.C., **69**, 371–377 (Oct. 1953).

Cotton Cellulose with reduced Crystallinity. L. Segal, M. L. Nelson, and C. M. Conrad. *Text. Research J.*, **23**, 428–435 (June 1953).

The variables in the application of ethylamine to cotton cellulose are investigated with reference to the effect on the final reduced crystallinity. The crystallinity is measured by the acid-hydrolysis residue method. Reduction of crystallinity is shown to take place rapidly and to be independent of the degree of polymerisation of the cellulose. The crystallite orientation is essentially unaffected by the treatment. A. B.

Effects of Ultrasonic Radiation on the Viscosity of Cellulose Solutions. R. F. Schwenker and J. C. Whitwell. *Text. Research J.*, **23**, 436–437 (June 1953).

Solutions of cotton and bright viscose rayon in cuprietylenediamine were exposed to ultrasonic radiation at a frequency of 400 kilocycles/sec. at a power output of 250 watts. The cotton samples showed a marked decrease in viscosity, whereas the viscose rayon samples were entirely unaffected. A. B.

Xanthation of Cellulose. II— Xanthation of Carbohydrates and of Cellulose. P. M. Cherkasskaya, A. B. Pakshver, and V. A. Kargin. *J. Appl. Chem. U.S.S.R.*, **26**, 311–320 (March 1953).

In model homogeneous systems used in kinetic studies of the xanthation of cellulose, the latter is replaced by sucrose and by alkali-sol., acid-degraded cellulose (of degree of polymerisation 82). Parallel heterogeneous experiments are carried out on the same cellulose preparation at a lower NaOH concn. The amounts of xanthate, Na_2S, and Na_2CS_3 are determined at intervals as reaction proceeds, and it is found that in all three cases the amount of xanthate formed rises to a max. and then falls. This behaviour is attributed to side-reactions already studied (see J.S.D.C., **69**, 209 (June 1953)), which disturb the esterification equilibrium by removing CS_2. The rate of xanthation in homogeneous medium $\propto [CS_2]^2$ when the degree of esterification <12–15%, and the heterogeneous reaction follows a first-order equation in its early stages. The rate of xanthation of sucrose falls with increase in NaOH concn., and the heterogeneous xanthation of hydrocellulose (carbohydrate : CS_2 : NaOH = 1·0 : 1·0 : 1·8) is, owing to the lower NaOH concn., somewhat faster than the homogenous reaction (carbohydrate : CS_2 : NaOH = 1·0 : 3·0 : 8·0). In the homogeneous reaction increase in temperature favours xanthation more than the side processes, but in the heterogeneous reaction this position tends to be reversed. A. E. S.

Mechanism of the Swelling and Dissolution of Xanthated Soda Cellulose Fibres— VIII. W. Schramek and L. Ahrberg. *Faserforsch. und Textiltech.*, **4**, 229–249 (June 1953).

An outline is given of previous investigations of this series, which originated from a consideration of the nature of fibre residues that interfere with the filtration of viscose. The work is now extended by further microscopical and ciné-microscopical studies of suitably stained pine pulp fibre, which is impregnated with 20% NaOH soln., aged, treated with CS_2, washed with 10% aq. Glauber's salt to arrest the chemical reaction and then allowed to swell in a weaker salt soln. The isolated fibres observed have a thin outer skin, which may be a part of the primary wall or an intermediate layer between the primary and the secondary walls; its rupture during swelling may lead to ballooning effects similar to those observable in the swelling of cotton. Below this skin (not above, as asserted by Lindtke (*Holzforsch.*, **4**, 65 (1950)) there is another, which may break away during swelling in the form of spiral ribbons. The actual effects observed depend on the duration of ageing and of CS_2 treatment. When both skins have broken away, the now unrestricted swelling of the secondary wall is accompanied by severe twisting of the fibre and extensive rupture along the now almost transverse spiral lines; the further breakdown of the fibre follows a regular but complex pattern. The discussion is illustrated by 41 photomicrographs. An exposition of the fine structure of the fibre is given, and there is a bibliography of work on the structure of cellulose fibres, particularly wood fibres (146 references).

A. E. S.

Fundamental Improvements in Regenerated Cellulose Fibre Structure. N. Drisch and L. Soep. *Text. Research J.*, **23**, 513–521 (Aug. 1953).

It is shown that, with particular cellulose xanthate dispersions, obtained from very uniform pulps with a high degree of polymerisation, it is possible to produce filaments of a remarkable resistance in the dry and also the wet state, showing also satisfactory elongation and fatigue-resistance. The elastic properties and work-till-rupture values are highly satisfactory. Dye uptake with Sky Blue 6B is more rapid than for high-tenacity rayon, although slightly slower than for cotton. A. B.

Cellulose Acetate from Raw Material to Haute Couture. D. Finlayson. *J. Textile Inst.*, **44**, P 307–P 327 (Aug. 1953).

An account of the development of cellulose acetate rayon and of the contributions to textile technology that have resulted therefrom. J. W. B.

The Wool Trade. K. G. Ponting. *J. Textile Inst.*, **44**, P 157–P 170 (May 1953).

An interesting account of the history of the English wool trade. J. W. B.

Heterogeneity of the Keratin Fibres. E. H. Mercer. *Text. Research. J.*, **23**, 388–397 (June 1953).

Hair and wool are shown to consist of keratinous and non-keratinous constituents, keratin being defined as a protein of the α-type stabilised by the cross-linkages formed by the disulphide bridges of crystine residues. An asymmetry found in wool is related to a difference in stability between one half of the cortex and the other half. The different responses of these to environmental changes may be responsible for the spontaneous twisting and curling of wool. A. B.

Tensile Behaviour of Some Protein Fibres. G. Susich and W. Zagicboylo. *Text. Research J.*, **23**, 405–417 (June 1953).

The tensile properties, including immediate elastic recovery, delayed recovery, and permanent set, of wool, mohair, human hair, casein, Vicara, and silk have been measured. A cycling technique was employed using the Instron tensile tester and the Sookne–Harris fibre extensometer. The effects of knotting and of mechanical conditioning are also investigated. It is shown that wool has an outstanding elastic recovery in the dry state, and that its elastic behaviour is improved after mechanical conditioning and when wet. Mohair and human hair, and to some extent casein and Vicara, have elastic properties similar to those of wool. The tensile behaviour of silk is different, being characterised by considerable permanent set and by high immediate elastic recovery after mechanical conditioning. A. B.

Crimping of Wool Fibres. M. Horio and T. Kondo; E. H. Mercer. *Text. Research J.*, **23**, 373–387 (June 1953).

Differential staining of wool under controlled conditions with basic dyes is employed to reveal a bilateral structure in wool fibres. This can be related to the crimping of the

fibres. Isolated fibres tend to coil rather than crimp, and in alkaline media a reversal of coiling direction occurs. In a comment, E. H. Mercer confirms the unsymmetrical structure. A. B.

Coiling of Wool Fibres immersed in Various Liquids. Y. E. Goldsworthy and W. R. Lang. *J. Textile Inst.*, **44**, T 230–T 244 (May 1953).

Standard lengths of fibre, lightly weighted, are immersed in various liquids, and their coiling characteristics studied. Merino 70s, Corriedale 56s, Lincoln 32s, Indian carpet wool, and cowhair are immersed in water, alkali, acid, methyl, ethyl, *n*-propyl, and *n*-butyl alcohols, diethyl ether, light petroleum, and benzene. Ardil and monofilament nylon are immersed in water; Merino 80s are given alternate water immersions and atmospheric dryings. Effects of cuticle abrasion, crimp, thickness, variation of test lengths, and permanent setting are studied. It is suggested that, when normally crimped fibres are suspended freely in cold water, they coil by the relaxation of torsional "cohesive" set and the release of axial twist; if the immersion is in hot water, the coiling includes also the relaxation of any torsional temporary set. Any rotation on drying is due to reassumption of axial twist alone. There is no evidence to support the theories that differential swelling or differential elasticity are significantly responsible for coiling. J. W. B.

Selective Absorption of Optical Antipodes by Proteins—II. W. Bradley and G. C. Easty. *J.C.S.*, 1519–1524 (May 1953).

The study of the selective absorption of (±)-mandelic acid on wool (*J.C.S.*, 499 (1951)) from an aq. soln. of (±)-mandelic acid is now examined more closely, and also extended to its *o*-, *m*-, and *p*-nitro, *p*-methoxy, and *o*-ethoxy derivatives, each of which is absorbed and resolved. The relation between the amounts of acid absorbed and resolved, and the stability of the wool protein–mandelic acid complexes, are determined. These results, together with parallel observations on the stability of the salts of mandelic acid with L-arginine and L-lysine, indicate the resolution of mandelic acid on wool to occur at the L-arginine and L-lysine residues, and mainly, if not entirely, in the crystalline regions of the fibre. The absorption of mandelic acid by wool is not accompanied by hydrolysis of the wool protein or by structural changes other than those which accompany salt formation. There is no evidence of resolution when degummed silk is brought into contact with aq. (±)-mandelic acid. L-Lysine, but not L-histidine or L-glutamic acid, resolves mandelic acid. H. H. H.

Modification in the Surface of Wool Fibres produced by Chemical Treatments—Electron-microscope Study. E. H. Mercer. *Australian J. Appl. Sci.*, **4**, 107–117 (1953): *Chem. Abs.*, **47**, 8377 (25 Aug. 1953).

Electron-microscopic examination of replicas of fibres before and after treatment show that halogens and sulphuryl chloride attack the scale edges and cracks; alcoholic KOH, in addition, causes pitting of the scales, indicating that the epicuticle is being attacked. C. O. C.

2-Aminoethanesulphinic acid—Intermediate in the Oxidation of Cysteine *in vivo*. J. Awapara. *J. Biol. Chem.*, **203**, 183–188 (July 1953).

Cysteine has been found to be converted in the liver of a rat into an amino compound, which has been isolated and tentatively identified as 2-aminoethanesulphinic acid. The mode of oxidation is probably a two-step one—(*a*) oxidation to cysteinesulphinic acid and (*b*) decarboxylation of the cysteinesulphinic acid. Proof of identity was furnished by comparison of the natural material with a synthetic sample. A procedure for the synthesis of 2-aminoethanesulphinic acid is described. P. G. M.

Relationship between Various Surface Properties of Wool Fibres. III—Sorption of Dyes and Acids in Wool Fibres. IV—Wettability. J. Lindberg. *Text. Research J.*, **23**, 573–584, 585–588 (Aug. 1953).

III—The diffusion rate of dyes and acids into wool fibres treated with alcoholic potash for different periods of time has been measured. It is shown that the epicuticle largely controls the diffusion rate. A theory is given in which the differences in permeability of the epicuticle are thought to be dependent on the polarity of the surface layer.

IV—It is shown that the wettability or the surface tension of wool fibres is dependent on the epicuticle, and that there are close relationships between friction, rate of diffusion of dyes and acids, and surface tension. It is also shown that the surface properties of the fibre can be changed without removing the epicuticle, and such change is thought to be due to an alteration of the molecular structure of the epicuticle. A. B.

Properties of Serges made from Blends of Wool with Acrylic-type Synthetics. H. Bogaty, N. R. S. Hollies, J. C. Hintermaier, and M. Harris. *Text. Research J.*, **23**, 536–544 (Aug. 1953).

Some preliminary results are given of a laboratory study of fabrics containing wool blended with varying amounts of the synthetic fibres based on acrylonitrile, viz., Orlon, dynel, and Acrilan. The properties studied include thermal resistance, surface hairiness, wicking properties, fabric stiffness, crease recovery, compression measurements, and shrinkage in laundering. A. B.

Absorption of Sodium Hydroxide by Protein and Polyamide Fibres in relation to pH Value. H. G. Fröhlich. *Deut. Textilgewerbe*, **52**, 502–504 (1950): *Chem. Abs.*, **47**, 8377 (25 Aug. 1953).

Silk, wool, nylon, Perlon U, Ardil, casein, and zein fibres were treated with aq. NaOH of varying concentrations for various lengths of time, and the amounts of NaOH absorbed determined by titration. The swelling of the fibres was also measured. The absorption curves for natural and regenerated protein fibres were similar. Beginning at about pH 7, the amount of NaOH absorbed increased slowly, until it became almost constant from pH 9·5 to 11·2. Beyond pH 11·5 it rose sharply, and then decreased again at pH >13. Curves showing the swelling of the fibres as the NaOH concentration increased were similar to the NaOH absorption curves. Swelling increased slowly from pH 7 to 11. Above pH 11 there was a sharp rise in the swelling curve, which was ca. 190% for wool, 95–100% for silk, 155–160% for casein fibres, ca. 40–45% for zein fibres, and ca. 100% for Ardil. Curves for the absorption of NaOH by nylon and Perlon U were almost horizontal from pH 8 to 11. Above pH 12 they rose sharply to a maximum NaOH absorption of 0·5–0·55 millimoles per gram of fibre. Several diagrams and a bibliography are given. C. O. C.

Milk Casein and Peanut Protein Fibres. R. L. Wormell. *J. Textile Inst.*, **44**, P 258–P 271 (July 1953).

An account of the nature and production of some regenerated protein fibres. J. W. B.

Technical Developments leading to Present Man-made Fibres. J. B. Quig. *Text. Research J.*, **23**, 280–288 (May 1953).

The historical background to synthetic fibres is reviewed from a chemical angle. The work of Carothers is discussed, and the various types of fibre-forming structures are listed. The most important properties of these polymers, such as linear symmetry, melting point, interchain attraction, and chain stiffness, are considered together with fibre morphology. A. B.

Current Problems and Future Trends in Synthetic Fibres. J. H. Dillon. *Text. Research J.*, **23**, 298–312 (May 1953).

The difficulties encountered with the synthetic fibres are discussed under the main headings of Fibre and Yarn Problems (uniformity, strength, elasticity, resilience, moisture absorption, dyeability, chemical stability, and surface characteristics), Fabric Problems (mechanical stability, soiling, ironing range, local melting, flammability, pilling, static electrification, wicking, abrasion, wrinkle resistance and crease retention, and tactile qualities). The various fibres are then considered in four groups—the inorganic fibres, the man-made cellulosic fibres, the man-made protein fibres, and the true synthetic fibres. A. B.

Grilon. H. Keller. *Textil-Rund.*, **8**, 410–414 (Aug. 1953).

Grilon is a polyamide fibre based on poly-ε-caprolactam and prepared by conventional methods by Fibron AG., Dormat/Ems. Two types are marketed: normal (4·5–5·5 g./denier, 20–26% extension at break) and high tenacity (6–7 g./denier, 15–18% extension at break). On being wetted, Grilon preserves 85–90% of its strength. Elastic recovery is almost complete up to 1–1·5 g./denier, but 0·1 g./denier should not be exceeded during processing

to avoid faults due to cockling because of the good recovery from stress. Regain is 4·2–4·5% at 20°C. and 65% R.H. Its m.p. is 215°C. and it becomes plastic above 165°C. Heat resistance is relatively good below 130°C. The density of Grilon is 1·13 g./c.c. It is essentially similar to other polyamide fibres. H. E. N.

Development of Terylene. J. R. Whinfield. *Text. Research J.*, **23**, 289–293 (May 1953).

The development of Terylene is traced from the laboratory work which preceded its discovery, through the pilot-plant stage, to large-scale production. A. B.

Polyvinyl Alcohol— Use as a Fibre and in Textile Finishes. J. A. Somers. *British Rayon & Silk J.*, **30**, (352), 65–67 (Sept. 1953).

An account of the manufacture of polyvinyl alcohol and of the manufacture of filament and staple fibres from it, with a special reference to Kuralon (Vinylon) made in Japan. The fibres are wet-spun, and rendered hydrophilic by treatment with formaldehyde. They can be dyed to medium depth with most dyes used for wool or cotton, but best results are obtained with disperse dyes. Kuralon fibres have sp. gr. 1·1–1·2 (nylon 1·14, cellulose rayons 1·52) and are thus suitable for soft bulky fibres. They can be hot-ironed satisfactorily, since they do not soften at 200°C. Fabrics made from these fibres retain their size and shape on washing, and the fibres have the same extensibility dry or wet, 3% regain at 65% R.H., and only 6·3% swelling in water. Brief reference is made to the use of polyvinyl alcohol and its derivatives in textile finishes. C. O. C.

Recent Investigations on Glass-fibre Fabrics. W. Bobeth. *Faserforsch. und Textiltech.*, **4**, 187–199 (May 1953).

Work on the effects of various agencies on glass fibres is reviewed (16 references), and investigations devoted to the verification and extension of this work on material of current production are described. Tables are given showing the changes in wet and dry strength produced by treatments with various agents; they reveal the considerable increases in wet strength produced by certain cationic agents and resins. Other tables show the effects of various treatments on the loss of strength produced by steaming, weathering, and rot-testing (burial). The effect of heat and the suitability of glass fibres for the reinforcement of concrete are among other subjects discussed. A. E. S.

PATENTS

Wet Spinning of Cellulose Acetate. BrC. *BP* 695,559

The point of convergence of filaments obtained by spinning upwards through a coagulating liquid is lowered by passing the yarn over a pulley above the surface of the liquid and fixed skew with respect to the natural plane of the yarn, so as to cause the yarn when it reaches the pulley to make contact with one side of the groove and later with the bottom of the groove. W. G. C.

Crimping Fibres. BrC. *BP* 696,761

A continuous sliver or tow of fibres is fed into one end of a confined passage, from the other end of which its emergence is strongly resisted, e.g. by a weighted hinged flap, thus subjecting the fibres to crimping pressure. They are either heated by an external source through the walls of the passage, or heated before being forced inside, e.g. by passing over a hot drum or by applying steam.

BP 696,762

To prevent fibres from clogging the hinge of the weighted flap, it is covered with a sheet of springy material, e.g. sheet metal, on the inside. J. W. B.

Protein Solutions. Eastman Kodak Co. *USP* 2,625,490

Protein, e.g. soyabean or feather protein, is dissolved or peptised in a 20–40% aq. soln. of an aromatic sulphonate of 6–8 C, e.g. Na toluenesulphonate. Such solutions do not foam. They may be used for the production of fibres and as finishes for paper or textiles. C. O. C.

Polyvinyl Alcohol Filaments. Kurashiki Rayon K.K. *BP* 697,097

Stronger filaments are obtained by causing the coagulating liquor to flow up a vertical tube and extruding the solution of polyvinyl alcohol into the coagulating liquor in the same direction and at a predetermined speed without imparting twist to the coagulated filaments. The polyvinyl alcohol solution must have a lower specific gravity than that of the coagulating liquor. This method also enables a much more compact apparatus to be used. C. O. C.

Fibre-forming Polyamides. Courtaulds. *BP* 697,332

Modification of *BP* 675,298, the chain length of the polypeptide being increased by heating the separated product *in vacuo* at 150–300°C. for 30–300 min. C. O. C.

Polyesters, Polyamides, or Polyesteramides of High Molecular Weight. Wingfoot Corpn. *BP* 697,334

Polyesters, polyamides, or polyesteramides containing several hydroxyl and/or amino radicals are treated with an *N*-acylpolyimide of a polycarboxylic acid of the type described in *BP* 681,054. The products are used for fibres, plastics, or films. C. O. C.

Acrylonitrile Copolymers. American Cyanamid Co. *BP* 695,449

Fibre-forming materials are obtained by polymerising a mixture of acrylonitrile (25–95 parts) and allyl or methallyl alcohol (75–5 parts). W. G. C.

Acrylonitrile Polymer Solutions. Chemstrand Corpn. *BP* 696,723

The viscosity of solutions of acrylonitrile polymers in organic solvents is reduced by adding 0·09–5·0% of nitric, hydrochloric, or sulphamic acid on the weight of the organic solvent. C. O. C.

Heat-treatment of Acrylonitrile Fibres. American Cyanamid Co. *BP* 695,448

Acrylonitrile fibres which have been spun, stretched, and dried can be heat-treated, whilst in a relaxed condition, for 0·1–5 sec. at 100–500°C., and are then shrink-resistant and have a greatly increased percentage elongation. W. G. C.

Methacrylonitrile Copolymers. BrC. *BP* 696,066

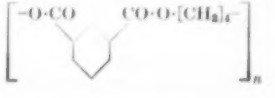
Copolymers of methacrylonitrile (55–95 parts) and vinylidene chloride (45–5 parts), soluble in acetone, are useful for the manufacture of filaments, films, etc. W. G. C.

Fibre-forming Polythioureas. Courtaulds. *BP* 696,964

Carbon disulphide is treated with a diamine whose amino groups are separated by a chain of > 2 C, and the resulting salt is heated in presence of water or steam until an intermediate polymer is precipitated. Most of the aqueous liquor is removed, after which heating is resumed until a fibre-forming polythiourea is obtained. This enables materials of low purity to be used. C. O. C.

Crystalline Tetramethylene *iso*Phthalate Polymer. Wingfoot Corpn. *USP* 2,623,034

Polyesters which can be cold-drawn to yield strong elastic fibres are obtained by condensing tetramethylene glycol with *iso*phthaloyl chloride. The product has the formula—



C. O. C.

Elastic Linear Copolymers. DuP. *USP* 2,623,033

Filaments having the high elastic recovery characteristic of rubber are obtained from the copolymer obtained by melting together compounds of formula $HOOC{\cdot}CH_2{\cdot}X{\cdot}CH_2{\cdot}COOH$ (X = chain of 4–9 atoms of which < 4 are O atoms and the remainder sat. hydrocarbon C atoms, any two O atoms being separated by > 1C atom, and there being < 4 C atoms as side-chain substituents) with terephthalic, dibenzoic, ethylenebis-*p*-oxybenzoic, tetramethylene-bis-*p*-oxybenzoic, or 2:6-naphthalic acid and with a polymethylene glycol containing 2–6 CH_2 groups. *USP* 2,623,031

Melt-blending of the above polymer with an aromatic polyester prepared by the melt polymerisation of one of the above dibasic acids with one of the polymethylene glycols results in a product which can yield filaments resistant to normal ironing. C. O. C.

Spinning Fibres from Rubber. Courtaulds. *BP* 694,869

In the production of rubber fibres by extruding a solution of rubber, containing a hydroperoxide as catalyst, into a coagulant bath containing sulphur dioxide, the presence of

water in the coagulant bath reduces the back-pressure caused by clogging of the jet-holes. W. G. C.

Addition Products of an Amine and Sulphone-activated Ethylenic Compounds (III p. 455).
Measurement of the Influence of Finishing Agents on Fibre Friction (X p. 471).
Papermakers' Felts (XI p. 473).
Manufacture, Structural Design, and Testing of Felts for the Papermaking and Allied Industries (XI p. 474).
Solubility in Methylene Chloride–Alcohol Mixtures of Fibrous Cellulose Acetate and the Products of its Partial Heterogeneous Hydrolysis (XI p. 474).

VII—DESIZING; SCOURING; CARBONISING; BLEACHING

Soiling and Soil Retention in Textile Fibres—Effect of Yarn and Fabric Structure in Soil Retention. W. T. Hart and J. Compton. *Text. Research J.*, **23**, 418–423 (June 1953).

The effect of yarn and fabric structure in soil retention is investigated, and it is shown that macro-occlusion, or interfibre and interyarn entrapment, of soil particles is a major factor in soil retention in all cases. It is also demonstrated that as the weight and the complexity of fabric structures increase, the number of macro-occluded soil particles increases. A. B.

Soiling of Acetate Rayon Carpets. F. Fortess and C.E. Kip. *Amer. Dyestuff Rep.*, **42**, 349–359 (8 June 1953).

The effects of oil content in the original cellulose acetate yarn, of scouring and dyeing procedures and auxiliaries used therein, and of jute oil content are studied with regard to the soiling of acetate rayon carpets, measurements of soiling being made photometrically after both floor tests and standard laboratory soiling. Clean acetate rayon fibre is intrinsically resistant to soiling, which is promoted by the primary lubricant, which should be kept to a minimum. Scouring and dyebath assistants are generally innocuous, but the usual carding and spinning oils cause severe soiling, which can be mitigated by using antistatic waxes. Oil in the jute backing migrates to the pile surface by wicking and causes serious soiling; this is successfully prevented by blocking with waxy lubricants or by using low-oil jute. Reagents employed are tabulated, the laboratory soiling test is described in detail, and results are illustrated photographically. J. W. B.

Colour Changes induced in Vat Dyeings by Organic Solvents. E. Kornreich. J.S.D.C., **69**, 297 (Aug. 1953).

Hydrogen Peroxide Bleaching of Textile Materials. H. G. Smolens. *Amer. Dyestuff Rep.*, **42**, P 408–P 410 (22 June 1953).

Cold bleaching, steam bleaching, and the white scour processes on cotton, wool, and rayons are surveyed, and their particular applicabilities discussed. J. W. B.

PATENT

Avoiding Corrosion of Stainless Steel when Bleaching with Chlorine Dioxide or Chlorites. M. Erlenbach and A. Sieglitz. *BP* 696,967

Corrosion of stainless steel by chlorine dioxide or chlorite solutions is avoided by adding to the bleaching bath an acid derived from nitrogen oxides and/or salts thereof, e.g. nitric acid or sodium nitrate. C. O. C.

Oxidising with Sodium Chlorite (VIII this page).
Fluorescent Brightening of Textiles (VIII p. 471).

VIII—DYEING

Becquerel Effect in the Presence of Dyes and the Action of Light on Dyes. P. J. Hillson and E. Rideal. *Proc. Roy. Soc.*, **A 216**, 458–476 (1953).

Photocurrent flows when a dye is adsorbed on an electrode and is illuminated. The direction of this current shows that the dye may be either oxidised or reduced. The primary step in reduction is direct transfer of an electron from the electrode to an excited dye molecule, and the primary step in oxidation is reduction of water to H atoms by excited dye molecules. The different effects found in photogalvanic experiments with dyes, in solution or adsorbed on the electrode, can be interpreted on this basis, and the significance of these reactions in the fading of dyes and tendering of textiles by light is discussed. Photosensitivity is shown as a function of the wavelength of the incident light for Fuchsin and Malachite Green. Within any one absorption band of the dye, the quantum efficiency of the photoreaction is independent of the wavelength. It is different for different absorption bands, and increases markedly in the ultraviolet. In acid solution, the photocurrent is positive and not reproducible. Curves are given for a group of Waxoline dyes of known composition. On the fibre, conditions are very similar to those in aqueous solution, as there is usually sufficient moisture adsorbed on the fibre to provide the OH radicals needed to react with the dye. Some OH radicals may be supplied by the fibre molecules; cellulosic fibres may do this, as they contain numerous OH groups. The resultant "cellulosic radicals" are less likely to react with oxygen than H atoms are, so that the reaction will not normally greatly increase the rate of fading, but the continual reduction and re-oxidation of the cellulose is likely to disarrange its structure and thus lead to tendering. C. O. C.

Common Sense on Colour in Textiles. C. M. Whittaker. *J. Textile Inst.*, **44**, P 237–P 246 (July 1953).

An entertaining survey of the problems and shortcomings of the dyeing and finishing industry is presented, with emphasis on the importance of research. J. W. B.

Fuel Efficiency in the Dyeing and Finishing Industries. W. Short. J.S.D.C., **69**, 329–335 (Sept. 1953).

Use of Non-ionic Level-dyeing Assistants with Chrome-complex Dyes. I. D. Rattee. J.S.D.C., **69**, 288–295 (Aug. 1953).

Fundamental Properties of Dyes. G. Otto. *Leder*, **4**, 1–8 (Jan. 1953): *Chem. Abs.*, **47**, 7779 (10 Aug. 1953).

The colour of a dye depends on the presence of double bonds and mesomerism. Dark dyes must have large molecules and many mesomeric forms, but this heteropolarity also increases their affinity for animal and vegetable fibres. Black dyes have greater affinity for chrome leather than yellow dyes, which have few mesomers. Only weak polar valency forces are involved; dyes react with vegetable fibres which contain no salt-forming groups. Ionic forces, however, draw dye and fibre together. If the dye contains many electrovalent groups, it will approach the hide fibre only to the range of the polar forces, and a readily hydrolysable compound will be formed. Polyamide fibres and collagen each take up decreasing amounts of dye as the number of sulpho groups in the dye increase. If, however, no electrovalent groups are present, the dye will reach the range of the non-polar forces and co-ordinate bonds will form. Chrome leather takes up not only more anionic dye but also more non-ionic dye than untanned hide, indicating that the additional binding of the dye to Cr is co-ordinate in nature. C. O. C.

Oxidising with Sodium Chlorite. G. O. Smith. *Amer. Dyestuff Rep.*, **42**, P 429–P 430, P 434 (6 July 1953).

The use of sodium chlorite (Textone) in carrying out various oxidising processes, including bleaching, oxidation of vat dyes, and the stripping of dyes (particularly on nylon) is discussed. J. W. B.

Dyeing of Viscose Rayon at High Temperatures. K. Butterworth. J.S.D.C., **69**, 362–370 (Oct. 1953).

Mechanism of the Dyeing of Nylon with Acid Dyes in Acid Baths. R. S. Pedersen. *Tidsskr. Textiltek.*, **11**, 82–85 (1953): *Chem. Abs.*, **47**, 7782 (10 Aug. 1953).

Study of existing theories, particularly as regards the ability of nylon to bind anions of dye acids, which increases rapidly with acidity of the dyebath to a supposed primary binding of the H ions, has confirmed the existing assumption. The binding seems to be of the nature of an adsorption complying with Freundlich's law. C. O. C.

Overdyeing of Nylon. C. D. O'Briain and R. H. Peters. J.S.D.C., **69**, 435–439 (Nov. 1953).

Interaction of Methyl Orange Anions with Lysine Polypeptides. D. B. Wetlaufer and M. A. Stahmann. *J. Biol. Chem.*, **203**, 117–126 (July 1953).

The precipitation of polylysine ($n = 130$) with methyl orange was shown to result in a rapid uptake of the dye over a small range of dye concentrations, this being characteristic of binding by precipitation. Polylysine was completely precipitated from solution before a stoichiometric amount of methyl orange was bound. This unsaturated precipitate continued to bind methyl orange as

the concentration of the latter was increased in what appeared to be an ion-exchange process. The solubility of the polylysine–methyl orange precipitate is strongly increased by neutral electrolytes. No evidence was found to indicate that methyl orange and polylysine form soluble complexes. The form of the dye precipitation curve was investigated as a function of polylysine chain length. Qualitative differences were found, and the possibility of applying precipitation curve analysis to other polyelectrolytes is discussed. P. G. M.

New Developments in the Dyeing of Orlon Acrylic Fibre. P. L. Meunier, J. F. Laucius, J. A. Brooke, and R. J. Thomas. *Amer. Dyestuff Rep.*, **42**, P 470–P 473 (20 July 1953).

Dyeing of Orlon with acid dyes by the Cu^+ method at atmospheric pressures and above, with disperse and with basic dyes, the piece dyeing of fabric, and the dyeing of blends of Orlon staple with wool and rayon are discussed. J. W. B.

Problems in Dyeing Synthetic-fibre Blends. E. Szlosberg. *Amer. Dyestuff Rep.*, **42**, P 431–P 434 (6 July 1953).

Methods of dyeing wholly synthetic fibres and their blends, at or near the boil, using swelling agents and cuprous ions, including the production of two-tone effects, are discussed. J. W. B.

Fluorescent Brightening of Textiles. A. Landolt. *Textil-Rund.*, **8**, 337–345 (July 1953).

A list of eleven Uvitex (Ciba) products, their properties, and the principles of their application are given. The measurement of the degree of brightening is discussed. At present there is no appropriate standard light source with the correct balance between ultraviolet radiation and visible light. With an ordinary ultraviolet lamp, the light emission of patterns treated with various fluorescent brightening agents is measured, and the effects of varying temp., time, and concn. of agent and of salt in application is illustrated by graphs. From the reduction in intensity of the fluorescence by a washing treatment at 40° C. for 30 min. with 5 g. of soap per litre, the percentage of agent removed from various materials is calc., with the following results— Uvitex RT: cotton 62·5%, wool 15·6%; Uvitex RS, cotton 88·8%; Uvitex NSJ, cotton 47·5%; Uvitex WS: wool 75%, nylon 18·75%; Tinopal 2B, cotton 55%; Leucophor B, cotton 47·5%; Blankophor B, cotton 45·3%. The fastness to washing of Uvitex RT on nylon and of Uvitex WS on acetate rayon is very good. H. E. N.

PATENT

Dyeing Acrylonitrile Polymers by the Cuprous Ion Method. Union Carbide & Carbon Corpn. *BP* 696,984

The materials are treated for ½ 15 min. at ½ 175° F. in an aqueous bath containing a water-soluble dye and cuprous ions. Good tinctorial value and excellent fastness to washing and rubbing are obtained with acid, direct cotton, and water-soluble acetate-rayon dyes. C. O. C.

Structure and Chemical Properties of Dyes in Relation to their Use (IV p. 457).

The Search for New Dyes in relation to Modern Developments in the Textile Field (IV p. 457).

Review of Textile Research and Development during 1952 (VI p. 466).

Relationship between Various Surface Properties of Wool Fibres. III—Sorption of Dyes and Acids in Wool Fibres. IV—Wettability (VI p. 468).

Colour Changes induced in Vat Dyeings by Organic Solvents (VII p. 470).

Avoiding Corrosion of Stainless Steel when Bleaching with Chlorine Dioxide or Chlorites (VII p. 470).

Relation Between Dyeing Properties of Wool and Modifications of the Fibre (X p. 472).

Reaction of Formaldehyde and Phenols with Wool. I—The Linking of Phenols. II—The Formation of Covalently Linked Mothproofing Agents. III—The Coupling of Linked Phenols to give Covalently Linked Dyes (X p. 472).

Ion-exchange Resins in the Textile Industry (XIII p. 476).

IX—PRINTING

Fundamental Processes of Textile Printing. V—Transfer of Disperse and Water-soluble Dyes to Cellulose Acetate during Steaming. E. H. Daruwalla and H. A. Turner. J.S.D.C., **69**, 240–254 (July 1953).

Fundamental Processes of Textile Printing. VI—Transfer of Disperse and Water-soluble Dyes to Nylon during Steaming. E. H. Daruwalla and H. A. Turner. J.S.D.C., **69**, 440–448 (Nov. 1953).

Novelty Printing Effects. H. P. Baumann. *Amer. Dyestuff Rep.*, **42**, P 463–P 465 (20 July 1953).

Certain printing procedures, not common in the United States, are described. J. W. B.

PATENTS

Pattern Effects. Heberlein & Co. *BP* 696,578

A precipitating agent is printed on the fabric, after which an aqueous dispersion of a natural or synthetic resin or a monomer or precondensate of a synthetic resin, natural or synthetic rubber, or a cellulose derivative is applied. This enables much more resin or the like to be applied than can be applied by normal roller printing. C. O. C.

Multilayer Colour-photographic Materials. Kodak. *BP* 696,474

Three superposed silver halide emulsion layers are used, that nearest the support being blue-sensitive and the other two layers being red- and green-sensitised silver chlorobromide emulsions in which the weight of bromide (calc. as AgBr) is 10–40% of the total silver halide. This gives maximum resolving power. C. O. C.

Multicoloured Photographs of Increased Colour Density. General Aniline. *USP* 2,623,822

In a multilayer colour film containing non-diffusing colour couplers it is possible to reduce the amount of colour-forming components in all the layers by one-third to half that normally required, and simultaneously obtain increase colour density, by repeating the colour development and bleaching steps two to four times, after removing the negatively developed silver image of a reversal film with acidic $K_2Cr_2O_7$ or $KMnO_4$, or by removing the unexposed silver halide of a negative or positive film by fixing. C. O. C.

Energisers for Aromatic Triamine Developers and Suppression of Proximity Development in Azine Colour Developers. General Aniline. *USP* 2,623,823

The tendency toward proximity development in the processing of tripacks by the azine method, when 2:4-diaminoaniline is used for colour development, can be wholly or nearly suppressed by using per litre of the azine developer 0·5–10·0 g. of the condensate of 2:4-diaminoaniline with a lower aliphatic α-ketocarboxylic acid or a lower aliphatic aldehyde. C. O. C.

X—SIZING AND FINISHING

Measurement of the Influence of Finishing Agents on Fibre Friction. H. L. Röder. *J. Textile Inst.*, **44**, T 247–T 265 (June 1953).

A method is presented in which fibre–fibre friction is measured by means of a torsion balance; reproducible results are obtained, for both static and dynamic coefficients of friction, on 50 mg. of fibrous material, and it is considered that the procedure is suitable for routine testing. The effects of various finishing agents on viscose rayon staple fibres are studied and described, and observations are made on the rheological phenomena of interfibre movement. Lubricating agents like mineral or vegetable oils have no special effect on fibres, and although they reduce interfibre friction to some extent, their lubricating power is limited. Introduction of ethylene oxide groups into the finishing agent favours hydrophilic properties, but also lowers the lubricating properties. Molecules substantive to the fibres may modify interfibre friction effects considerably, particularly with regard to static friction. J. W. B.

Use of Resins and Plastics in Textile Finishing. H. H. Mosher. *Amer. Dyestuff Rep.*, **42**, P 402–P 406, P 410 (22 June 1953).

Chlorine Retention in Textile Finishes. A. H. Wachter, S. Davison, and M. K. Knight. *Amer. Dyestuff Rep.*, **42**, 261–263 (27 April 1953).

A proposed accelerated chlorine test, comprising exposing to 0·17% available chlorine at 140°F. for 10 min., rinsing, drying, and heat-treating at 400°F. for 30 sec., is applied to experimental melamine–formaldehyde resin-finished rayon fabrics, and is compared with strength losses caused by 25 repeated launderings which include 0·008% Cl in the third step. Strength losses are lower than those in the accelerated test. Pressing at 275°F. does not increase the loss; 80-hr. exposure in the Fadeometer following the launderings greatly increases the losses, but still not to the level of the accelerated test. It is concluded that light activates the degradation, and that high strength-loss in fabrics containing certain finishes is due to ageing after chlorine adsorption during laundering.

J. W. B.

Chlorine-retentive Properties of Rayon Fabrics treated with the Melamine Type of Rayon Finish—Strength Changes in the Rayons. G. S. Wham and P. B. Mack. *Amer. Dyestuff Rep.*, **42**, 285–295 (11 May 1953).

Melamine–formaldehyde-finished viscose rayon fabrics are tested by repeated launderings, including chlorine treatments, hot pressings, and exposures in a Fadeometer. Resin-treated fabrics show greater discoloration than controls after repeated hot-pressing; the strength deteriorates more, particularly as washing progresses; light is an important factor in increasing the strength loss. It is clear that chlorine, in the small controlled amounts used in laundering, is retained by these fabrics, and contributes to their loss in strength.

J. W. B.

Chlorine Retention of Resin-finished Rayon Fabrics—Strength Changes in Rayon-finished with Formaldehyde Resins. G. S. Wham and P. B. Mack. *Amer. Dyestuff Rep.*, **42**, 328–334 (25 May 1953).

Rayon gabardines, untreated and treated with urea–formaldehyde resin, are subjected to laundering treatments with and without addition of chlorine during one stage, which are varied by including steam pressing and exposure in a Fadeometer and to light through window glass. In the early stages resin-treated fabrics sometimes degrade less than the controls, probably owing to the protective action of the resin, which has absorbed insufficient chlorine to cause degradation. Low-temperature ironing causes no significant increase in strength loss, but light has a considerable effect in promoting degradation.

J. W. B.

Preliminary Steps in the Development of an Accelerated Laboratory Test for Chlorine-retention Properties in Resin-treated Fabrics. G. S. Wham and P. B. Mack. *Amer. Dyestuff Rep.*, **4**, 360–362 (8 June 1953).

Two fabrics, each having two colours alternating with white areas, are finished with and without a melamine–formaldehyde aftertreatment, and repeatedly laundered with and without a chlorine addition, and with intermediate Fadeometer exposures and hot pressing. Differences in colour fastness are generally not so great as the previously reported strength differences, but where colour loss does occur, apparently owing to chlorine retention, it is greater with those fabrics containing resin.

J. W. B.

Preliminary Steps in the Development of an Accelerated Laboratory Test for Chlorine-retention Properties in Resin-treated Fabrics. G. S. Wham. *Amer. Dyestuff Rep.*, **42**, 389–393 (22 June 1953).

Viscose rayon fabrics are given urea–formaldehyde and melamine–formaldehyde finishes and are subjected, in the laboratory, along with untreated controls, to chlorination (NaOCl) followed by hand-pressing with an iron at 400°F. Resin-treated fabrics show a greater strength loss, though pressing does not appear to be a contributory factor; yellowing of white areas is, however, more marked. The method used would seem to be suitable as an accelerated laboratory test for chlorine retention, although strength losses are smaller than in laundering trials with intermittent light exposure.

J. W. B.

Further Steps in the Development of an Accelerated Laboratory Test for Chlorine-retention Properties in Resin-treated Fabrics. G. S. Wham and R. N. Lynn. *Amer. Dyestuff Rep.*, **42**, 421–424, 441 (6 July 1953).

Rayon fabrics finished with melamine resin are, along with controls, subjected to three different chlorine treatments, after which one set of samples is stored in the dark prior to testing, and the others are exposed for three different periods in a Fadeometer. At a low chlorine concentration (0·015%) no consistent trends are noticed in the strength loss of the resin-finished fabric as compared with the control; at a concentration of 0·13% the loss in strength is consistently greater than that of the control; and at 0·5% concentration both types are considerably degraded. At the two higher concentrations there is a general loss of strength with increasing light exposure, the more so with the resin-treated fabrics, which show also more yellowing.

J. W. B.

Polymerisation of a Series of Vinyl Monomers in Wool. N. K. Boardman and M. Lipson. *J.S.D.C.*, **69**, 335–338 (Sept. 1953).

Review of Mildewproofing Treatments. E. C. Hansen and C. A. Bergman. *Amer. Dyestuff Rep.*, **42**, P 466–P 468 (20 July 1953).

Tests with D.D.T. Non-ionic Emulsion for Mothproofing Washable Wool Goods. H. Laudani. *U.S. Bur. Entomol. & Plant Quarantine* E-858, 15 pp. (1953): *Chem. Abs.*, **47**, 7725 (10 Aug. 1953).

Wool cloth absorbed more D.D.T. from dilute non-ionic emulsion than was expected by theory. Presence of soaps, detergents, and water-softening agents had no noticeable effect on the rate of absorption of the D.D.T. by the wool. The amount of D.D.T. taken up by the wool varied from 5·9 to 54·0% of that present in the emulsion, depending on the type of material treated, type of treatment, and the concentration of D.D.T. in the bath. As little as 0·08% of D.D.T. on the wool prevented damage by carpet-beetle larvæ and as little as 0·1% that by clothes-moth grubs.

C. O. C.

Aminisation of Cotton. W. A. Reeves and J. D. Guthrie. *Text. Research J.*, **23**, 522–527 (Aug. 1953).

Methods are described for the aminisation of cotton in the form of raw stock, sliver, yarn, and fabric by curing with 2-aminoethylsulphuric acid in the presence of sodium hydroxide. Other methods of aminisation are discussed.

A. B.

Chemical and Physical Properties of Aminised Cotton. W. A. Reeves, O. J. McMillan, and J. D. Guthrie. *Text. Research J.*, **23**, 527–532 (Aug. 1953).

The chemical reactions of aminised cotton are shown to be quite different from those of untreated cotton. The aminised cotton absorbs cotton dyes very rapidly, and direct dyes in particular are more resistant to light and laundering on aminised cotton. Acid dyes are also absorbed by aminised cotton but are not fast to laundering. Treated fabric may be given considerable rot-resistance by reaction with 8-hydroxyquinoline-5-sulphonic acid followed by chelation with copper.

A. B.

Relation Between Dyeing Properties of Wool and Modifications of the Fibre. G. Lagermalm, J. Lindberg, and L. Weissbein. *Text. Research J.*, 23, 398–400 (June 1953).

The effect of steaming on the rate of dyeing of solvent-extracted wool is determined. It is shown that the rate is not altered on steaming undamaged or surface-treated wool, but that if the wool is treated in aqueous alkaline solution the dyeing time is increased by steaming. This indicates that the effect of steaming is due to modifications in the bulk of the fibre.

A. B.

Reaction of Formaldehyde and Phenols with Wool. I—The Linking of Phenols. II—The Formation of Covalently Linked Mothproofing Agents. III—The Coupling of Linked Phenols to give Covalently Linked Dyes. K. G. Johnson. *Text. Research J.*, **23**, 443–448, 449–457, 457–462 (July 1953).

I—It is shown that the reactions of phenols and formaldehyde with wool keratin give greater weight increases than the corresponding reactions with the phenols alone, and that this is not due to the deposition of

polymers. The weight increase is stable to subsequent alkaline hydrolysis or extraction with organic solvents, but is readily removed by warm acid hydrolysis. It is probable that the phenols are covalently linked by methylene bridges to the ε-amino groups of lysine.

II—Synonymous with the weight increase described above is a resistance to the attack of clothes-moth larvæ. The stability of this moth proofing is identical with that of the weight increase.

III—It is shown that phenols which are covalently linked to wool keratin may be coupled with diazonium compounds to give covalently linked dyes. With all the phenols and amines examined, better results are produced if the initial treatment is carried out in the presence of formaldehyde, the best results being obtained with 3-hydroxy-2-naphthoic acids and its arylamides. Good colorations are obtained, which are fast to subsequent alkali washing. A. B.

Reaction of Polyacrylic Acid with Nylon. A. C. Nuessle and R. F. Crawford. *Text. Research J.*, **23**, 462–468 (July 1953).

It is shown that polyacrylic acid can be heat-set on nylon to give a very durable, crisp finish, the durability being proportional to the severity of cure. It is suggested that actual chemical combination between the polyacrylic acid and the nylon occurs, although the reaction mechanism is not elucidated. The addition of glycerol increases the durability at a given cure. The properties of the treated fabric are outlined. A. B.

PATENTS

Rendering Loose, Raw Cellulose Fibres. Flame- and Fire-resistant. Lockport Cotton Batting Co. *BP* 696,975

The raw fibres are treated in a bath containing a flame- and flash-resistant agent, e.g. borax and boric acid, and a wetting agent. C. O. C.

Composite Textile Material comprising a Felted Layer. N. P. F. Sommer. *BP* 694,460

Through a felt backing are passed tufts of fibres, which are drawn partly to the outside of one surface, on which a layer of adhesive is then placed to amalgamate and hold the tufts firmly, to increase the toughness of the felt, and to fix a covering material which is subsequently applied. J. W. B.

Bonded-fibre Fabrics. British Cellophane. *BP* 695,805

Bonded-fibre fabrics with improved lateral strength are made by laying a carded web with the fibres disposed longitudinally and applying to it one or more carded webs cross-laid at an angle and each doubled back repeatedly at the edges of the first web, the respective speeds of delivery being adjusted to form a layer of uniform thickness. The whole is bonded together; if the webs are of viscose rayon staple the bonding agents is viscose, e.g. a solution containing 6% of NaOH and 8·5% of cellulose, containing pigments or dyes if desired, and regeneration is effected by aftertreating the impregnated webs in the usual way. J. W. B.

Finishing Cellulosic Textiles with Urea Divinyl Sulphone Addition Products. American Viscose Corpn. *USP* 2,623,807

The material is impregnated with an aqueous liquor containing the precondensate of urea and a sulphone-activated ethylenic compound, e.g. divinyl sulphone, drying and baking to cure the resin. An alkaline catalyst, e.g. NaOH, is preferably present. The finished material is dimensionally stabilised to repeated washing. C. O. C.

Resin Finish on Cellulosic Fibres. Joseph Bancroft & Sons Co. *USP* 2,622,995

Fabric composed of natural and/or regenerated cellulose is impregnated with 70–90% on its dry weight of an aqueous solution containing 5–20% by wt. of resin-forming materials, 1–4% of a permanent softener, and 3–5% on the weight of the resin-forming materials of a delayed-action catalyst, e.g. carbazide hydrochloride, dried at 200–350°F. until it contains its natural regain, cooled, given a mechanical finish, e.g. calendered, schreinered, or embossed, at <200°F., and then baked to set the resin. This imparts a lustrous finish without loss in handle or porosity. A considerable degree of crease resistance is also imparted. C. O. C.

Linen-like Finish on Regenerated Cellulose Staple. Joseph Bancroft & Sons Co. *USP* 2,622,994

Fabrics made from regenerated cellulose staple and 0–50% of cotton are impregnated with an aqueous solution of a thermosetting resin precondensate, dried to 10–15% moisture content, quickly cooled to 60–100°F., passed through a chasing calender at low temperature to avoid formation of glaze, and finally baked to set the resin. A linen-like finish fast to washing is obtained together with crease resistance, increased strength, and resistance to spotting and soiling. C. O. C.

Resin-impregnated Wool Felts, Blankets, etc. Monsanto. *USP* 2,622,996

Heavy wool fabrics are given a resin finish without impairing their resilience and stiffness by impregnation with a solution or dispersion containing a thermosetting resin and a polyhydric aliphatic mercapto alcohol, e.g. 2-mercaptoethanol, followed by baking to set the resin. This imparts improved wet and dry strength and resistance to shrinkage and water absorption. C. O. C.

Water-repellent Finish. Dynamit-AG. vormals Alfred Nobel & Co. *BP* 697,252

Natural or artificial textiles are given a water-repellent finish and a silken handle by impregnating them with an aqueous solution of an *N*-glycoside of an aliphatic amine of >11 C, e.g. prepared from glucose and heptadecylamine, and heating the wet materials to <60°C. to split the *N*-glycoside with separation of the sugar, which is subsequently washed out. The liberated amine subsequently becomes converted into the insoluble carbamate by the following reactions—

$$R\cdot NH_2 + CO_2 \rightarrow R\cdot NH\cdot COOH$$

$$R\cdot NH\cdot COOH + R\cdot NH_2 \rightarrow R\cdot NH\cdot COO\}NH_3\cdot R$$

C. O. C.

Coating Bandage Material. Firma Lohmann. *BP* 697,145

Bandage material is coated or impregnated with a vehicle to which there has been applied a pulpy mass, preferably consisting of pancreas ferments, dried bile, sodium bicarbonate, penicillin, and a solvent and binding agent. The treated material is dried at 45°c, under reduced pressure. The coating composition is produced by the aid of sterile water at >5°c. Methyl cellulose is preferably used as the binder and sterile water as the solvent. C. O. C.

Surfaced Fabric. Universal Moulded Products Corpn. *USP* 2,625,499

The fabric is impregnated with one type of resin and then coated with another type of resin. This enables resins to be used as the coating which would not normally have sufficient adhesion to the fabric. C. O. C.

Linoleum. Sloane-Blabon Corpn. *USP* 2,624,068

Granulated linoleum composition is fed to a calender whose rolls have the same speed and are continuously washed or cleaned, so that they present fresh, somewhat lubricated surfaces to the composition. This allows the particles to be free to adjust their position with respect to the rolls as they approach or pass through the nip, with a consequent minimum of distortion or stretching of the particles. This results in a product having a granite-type design. C. O. C.

Aqueous Emulsions of Organic Silicates—Non-slip Finish (III p. 455).
Mechanism of the Formation of Electrostatic Charges on Fibres (VI p. 466).
Cotton Cellulose with reduced Crystallinity (VI p. 467).
Fuel Efficiency in the Dyeing and Finishing Industries (VIII p. 470).
Coating Webs (XI p. 475).

XI—PAPER AND OTHER CELLULOSIC PRODUCTS

Papermakers' Felts. R. Collins. *Proc. Tech. Sectn. British Paper & Board Makers' Assocn.*, **34**, 33–46 (Feb. 1953).

The manufacture, washing, and wetting of felts and their weights and weaves for paper and board machines and MG cylinders are discussed as well as the application of nylon, resin treatment, and tanning. S. V. S.

Manufacture, Structural Design, and Testing of Felts for the Papermaking and Allied Industries. E. Race. *J. Textile Inst.*, **44**, P 406–P 432 (Aug. 1953).

Relative Reactivity of the Hydroxyl Groups of Cellulose. T. E. Timell. *Svensk Papperstidning*, **56**, 483–490 (15 July 1953).

From a review of the literature it is concluded that the 6-hydroxyl group is generally the most reactive, though in some cases conditions favour the preferential conversion of the 2-hydroxyl group; and the 3-hydroxyl has always been found least reactive. A series of ten different low-substituted carboxymethyl celluloses and the water-soluble fraction of each were analysed in a study of the relative reactivity of the primary and secondary OH groups during carboxymethylation. The primary OH groups were found to be approx. twice as reactive as the average of each of the secondaries. Over oxidation occurs in periodate oxidation, as compared with the periodate–formaldehyde method, probably accounting for the low content of the secondary OH groups found by previous investigators. As the OH groups of cellulose apparently behave differently towards different reagents and under different external conditions, general statements regarding their relative reactivities accordingly seem uncertain.

S. V. S.

Thermal Depolymerisation of Cellulose. D. Tishchenko and T. Fedorishchev. *J. Appl. Chem. U.S.S.R.*, **26**, 393–396 (April 1953).

Cotton cellulose begins to dissolve in water at an appreciable rate at ~235° C., forming highly reducing products. When it is heated dry (in a stream of Hg vapour), rapid decomp. commences at 250° C. The cotton used in this work is first given the very mild alkaline treatment that was shown by Ermolaeva (*ibid.*, **21**, 543–547 (May 1948)) to be necessary for the removal of the acetyl groups present in wood pulp which give rise to acetic acid at high temp., thus causing hydrolytic decomp. of the cellulose. Some earlier thermal-stability work on cotton cellulose is criticised on the grounds that no deacetylation treatment was given. No direct comparison is made between untreated and "deacetylated" cotton.

A. E. S.

Effect of Daylight on the Periodate Oxidation of β-Methyl Glucoside, β-Methyl Cellobioside, and Cellulose. F. S. H. Head. *J. Textile Inst.*, **44**, T 209–T 223 (May 1953).

The periodate oxidation of β-methyl cellulose, β-methyl cellobioside, and cellulose is studied in presence and absence of light. It is considered doubtful whether the initial (Malaprade) stage of the oxidation is affected, but the rates of subsequent "over oxidation" reactions are greatly increased by light, there being no detectable discontinuity in the rate curves at this point. Over oxidation does, however, proceed in the dark, ultimately with the same result as with light-accelerated reactions. With sufficient periodate the organic matter is oxidised to CO_2 and H_2O; otherwise reduction of periodate to iodate is followed by reduction of iodate and liberation of iodine. With a moderate excess of periodate over that required for the Malaprade reaction, cotton cellulose suffers a progressive loss of weight in daylight, but in the dark the loss is not considerable until the Malaprade stage is passed. Oxidation beyond this point leads ultimately to a homogeneous, non-viscous solution. More formaldehyde and formic acid are formed than would be expected from terminal glucose oxidation, the yield of acid being higher with than without light. For a given degree of oxidation, periodate oxycelluloses prepared in daylight have lower copper numbers than those prepared in the dark, and it is suggested that the ratio of Malaprade to other types of oxidation is higher in the dark than in daylight. The mechanism of cellulose over oxidation is also discussed.

J. W. B.

Moisture Regain of Methyl Cellulose and Cellulose Acetate. G. C. Gibbons. *J. Textile Inst.*, **44**, T 201–T 208 (May 1953).

Results obtained on the moisture regains of methyl celluloses and cellulose acetates of varying degrees of substitution are explained on the basis that the regain of cellulose is affected by substituent groups, by the degree of substitution, by the proportion of OH groups accessible to water among those remaining unsubstituted, and by the possibility that trisubstituted derivatives may also crystallise and further reduce the already low water absorption. Measurements are made at 65% and 80–85% R.H.; expressed as molecules of water per glucose residue, the regain increases initially, and then decreases linearly with increasing degree of substitution. The linear relation is considered to show the effect of substitution on the hygroscopicity of fully accessible cellulose, and when extrapolated to zero degree of substitution the intercept represents the proportion of water in fully accessible unsubstituted cellulose. For any specimen of cellulose the ratio of observed moisture regain to the regain of fully accessible cellulose at the same R.H. gives the proportion of OH groups accessible to moisture. For bleached cotton, mercerised cotton, and viscose rayon the values are ca. 0·4, 0·5, and 0·8 respectively.

J. W. B.

Effect of a Wetting Agent on the Hydrolysis of Highly Methylated Cotton Cellulose. R. E. Reeves and B. J. Barrett. *Text. Research J.*, **23**, 510–512 (July 1953).

It is shown that the rate of hydrolysis of highly methylated cotton cellulose is markedly increased by the addition of a suitable wetting agent. The wetting agent did not alter the minimum viscosity of the undissolved residue, but it greatly shortened the time of hydrolysis necessary to arrive at the minimum value.

A. B.

Effect of Salts on Solutions of Ethyl Hydroxyethyl Cellulose. I. Jullander. *Svensk Papperstidning*, **56**, 443–450 (30 June 1953).

Ethyl hydroxyethyl cellulose can be salted out from an aqueous solution, the salting-out properties of salts being expressed quantitatively as flocculation points, determined by a method resembling the customary salt-point measurements in the viscose industry. The flocculation points were determined for about seventy salts and three types of ethyl hydroxyethyl cellulose, and a comparison was made with commercial methyl celluloses. The effects of mixtures of Na_2SO_4 and $NaNO_3$ were investigated, and it was not found possible to predict the behaviour of a salt mixture from measurements on the single components. Possible relations between flocculation point and hydration numbers or hydration energies of the salts and between flocculation point and activity of water are discussed. It was found, in studies of a few salts, that viscosity passes through a pronounced maximum, whilst with increasing salt concentration turbidity changes little up to the flocculation point.

S. V. S.

Solubility in Methylene Chloride–Alcohol Mixtures of Fibrous Cellulose Acetate and the Products of its Partial Heterogeneous Hydrolysis. F. S. Sherman and P. V. Kozlov. *J. Appl. Chem. U.S.S.R.*, **26**, 524–531 (May 1953).

Various mixtures of methylene chloride with methanol and with butanol are examined as solvents for fibrous cellulose triacetate (I) (mol. wt. ~132,000; 61·75% combined acetic acid) and two of its partial-hydrolysis products (II and III) (60·12% (II) and 56·6% (III) combined acetic acid), obtained as already described (see J.S.D.C., **68**, 330 (Aug. 1952)); viscosity measurements are made on the solutions obtained. I and II are almost identical in behaviour and are of good solubility in all mixtures examined, but III is soluble only in a narrow range of methylene chloride–methanol mixtures and gives highly viscous solutions. Mixtures containing 90–80% of methylene chloride and 10–20% of methanol are the best general solvents for cellulose acetate preparations varying widely in chemical composition.

A. E. S.

Fractionation as a Method of Studying the Uniformity of Carboxymethyl Celluloses. T. E. Timell. *Svensk Papperstidning*, **56**, 311–323 (15 May 1953).

The chemical uniformity of several carboxymethyl celluloses has been studied by fractionation procedures. Fractionational precipitation was found to take place according to both chemical constitution and molecular weight, and showed a technical carboxymethyl cellulose to be rather non-uniform in both these respects. A summative fractional solution procedure employing aqueous methanol solutions gave fractions varying more in degree of substitution than in molecular weight. The non-uniformity of the samples studied is interpreted in terms of longer or shorter sections of unchanged chain

molecules still kept more or less firmly together by hydrogen bonds, but a direct proof of the existence of such localities in carboxymethy celluloses with poor solubility properties would be desirable. S. V. S.

Decomposition of Lignin by Metallic Sodium in Liquid Ammonia — V. A. F. Semechkina and N. N. Shorygina. *J. Gen. Chem. U.S.S.R.*, **23**, 593–595 (April 1953)

Previous investigations in this series (see J.S.C.I., **67**, 46 (Jan. 1951)) were concerned with fir lignin. Pine lignin is now examined in a similar manner and yields 18% of ether-sol. material, from which dihydroeugenol and 1-(4-hydroxy-3:5-dimethoxyphenyl)propane are isolated. A. E. S

PATENTS

Felted Vegetable-fibre Web Material. ICI. *BP* 696,891

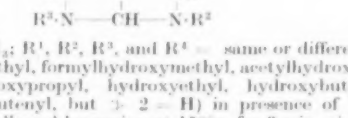
Vegetable fibre suitable for the manufacture of felted web material is suspended in water free from aluminium salts and containing a water-soluble salt of a carboxyalkyl cellulose, e.g. a sodium carboxymethyl cellulose, and a water-soluble sodium salt of a phosphorus oxy-acid of which the anhydride is P_2O_5 and which prevents precipitation of aluminium soaps in water, e.g. sodium hexametaphosphate. The amount of the phosphate is sufficient to prevent precipitation of the aluminium salt of the carboxyalkyl cellulose on the subsequent addition of an aluminium salt to the resulting suspension, so that dispersed rosin which has been included at any stage prior to the felting operation is precipitated. S. V. S.

Wet-strengthened Absorbent Paper by Glyoxal Treatment. A. P. W. Products Co. *USP* 2,622,960

The paper is treated with an aqueous solution of glyoxal, dried to 3–7% moisture content and then heated at 4–212° F. to cause the glyoxal to react with the cellulose. C. O. C.

Paper of High Wet Strength. Mathieson Chemical Corpn. *USP* 2,624,686

Incorporation of 0·5–10·0% (in the dry wt. of the paper) of a compound of formula—



(X = H or CH_3; R^1, R^2, R^3, and R^4 = same or different H, hydroxymethyl, formylhydroxymethyl, acetylhydroxymethyl, hydroxypropyl, hydroxyethyl, hydroxybutyl, and hydroxybutenyl, but ≯ 2 = H) in presence of an acid catalyst followed by curing at 150° C. for 3 min. gives high wet strength, high water absorptivity, and good resistance to oil and other liquids. C. O. C.

Coating Paper. Inland Wallpaper Co. *USP* 2,622,558

In a machine for coating wallpaper before it is printed the paper passes through a nip formed by a backing roll and the colour roll to receive the coating composition from a fountain roll mounted on a colour pan. The colour pan and the fountain roll are movable as a unit so that when the machine is stopped the fountain roll can be dropped away from the backing roll. C. O. C.

Paper-coating Compositions. Kodak. *BP* 696,496

A dispersion of barium sulphate in water for use in photographic paper-coating compositions contains as peptising agent for the barium sulphate 0·33–1·0% on the wt. of sulphate of product resulting from the treatment of a synthetic resin containing carboxylic anhydride groups with ammonia or a primary or secondary aliphatic amine. The synthetic resin may contain >10 recurring units in the molecule, the amine may contain alkyl groups of ≯6 C, and the peptising agent may be an ammoniated copolymer of styrene and acrylic or maleic anhydride, or of vinyl acetate and maleic anhydride. The aqueous dispersion is mixed with a colloid binder, e.g. gelatin and a styrene–butadiene latex, and the resulting composition applied as a layer to the paper surface and then dried. S. V. S.

Carbon Paper. Günther Wagner. *BP* 697,307

Untreated carbon tissue paper, dyed in the beater with a colour other than black is coated with a thermoplastic artificial resin in which non-translucent pigments, preferably soot, are incorporated, which screen off light but the colour of which is not transferred with the normal carbon coating. A carbon-paper colouring layer is then applied in the usual manner, over the resinous layer. S. V. S.

Paper and Paperboard. Dow Corning. *BP* 697,240

Paper or paperboard is rendered non-adherent to normally adherent organic materials by coating with a methyl hydrogen polysiloxane, end-blocked with trimethylsiloxy units, and of general formula

$$(CH_3)_3SiH_bO_{(1-a-b)/2}$$

(a = 1·1–1·5; b = 0·75–1·25; $a + b$ = 2·0–2·25). S. V. S.

Superficially Saponified Films or the like of Cellulose Esters of Fatty Acid. Philips' Gloeilampenfabrieken. *BP* 696,103

Foils, films, or the like of fatty acid cellulose esters of fatty acids having 1·5–3 mol. of fatty acid per glucose residue are saponified in a bath containing an alkaline substance and an organic solvent, and after saponification has proceeded to the desired depth, the foil is aftertreated in one or more baths to terminate the saponification. At least one aftertreatment bath, which does not comprise an aqueous solution of a mono- or a di-basic acid, contains a non-ionogenic agent with the property of reducing aqueous swelling of partly esterified cellulose having 0·5–1·5 mol. of fatty acid per glucose residue, e.g. a solution of a salt of a sulphated aliphatic hydroxycarboxylic acid of >9 C. One aftertreatment bath may contain a solvent for the plasticiser of the cellulose ester, and both non-ionogenic and ionogenic agents may be present. One aftertreatment bath may contain a saturated primary alcohol, e.g. *n*-butanol, having a main chain of 4–11 C. Immediately after saponification an aftertreatment bath is used containing a solution of a polybasic acid, e.g. phosphoric acid in *n*-butanol. An aftertreatment bath may contain an ionogenic agent and a substance with 3–6 alcoholic hydroxyl groups in the molecule, a water-soluble salt of an organic carboxylic acid having an equivalent weight ≯100, and an organic carboxylic acid, readily soluble in water, dissolved in the alcohol. The salt of a polybasic acid, at least tribasic, may be used, and an aqueous fluoride solution may be employed. A bath may contain toluene and/or tetrahydronaphthalene and/or benzene and/or a liquid ether. Light-sensitive substances for the production of metallic photographic images, adapted to production by physical development, may be incorporated in the foil. S. V. S.

Surface Treatment of Regenerated Cellulose Film. British Cellophane. *BP* 695,555

The tendency of sheets of film to stick together is reduced by treatment with an aqueous dispersion of wax, the wax particles carrying an electric charge of opposite sign to that on the film. W. G. C.

Coating Webs. Kodak. *BP* 694,570

A coating of controlled thickness, e.g. of photographic emulsion, is applied to a travelling web, e.g. a film base, by passing it through a coating apparatus in which it receives on one side sufficient coating to give at least 50% excess thickness, and then passing the coated material through a substantially vertical wedge space formed by two nearly parallel flat surfaces, one of which abuts the non-coated surface of the web and is parallel to the plane of movement of the web, and the other of which is manually adjustable and held at a predetermined position, so that (*a*) the exit slit of the wedge is approximately equal in thickness to the thickness of the web plus about 1½ times the desired thickness of the coating liquid, and (*b*) the angle of the adjustable surface is such that the plane of the surface of the liquid of the web coming from the coating apparatus cuts the plane of the said adjustable surface at a distance from the exit slit in a direction against the motion of the web at least 50–1000 times the desired thickness of the coating. S. V. S.

Coated Paper. United States Rubber Co. *BP* 696,118

Paper is coated with a thin, continuous, flexible, transparent layer of a resinous copolymer of styrene and acrylonitrile (65–80% styrene, 35–20% acrylonitrile), or of mixtures of 50–95% of the resinous copolymer and 50–5% of a rubbery copolymer of 1:3-butadiene and acrylonitrile. The highly glossed film does not separate from the paper when pressure-sensitive adhesive tape is applied firmly to the coated surface and then pulled suddenly, but only the body of the paper splits. The amount of coating ranges from 0·1 to 0·5 lb. of polymeric material per 1000 sq. ft. of

coated surface, and the coating is a hot-calendered film, formed *in situ*, of the solids deposited by drying from an aqueous dispersion of the polymeric material. The bond with the surface of the paper is due to the absorption of ~20% by weight of the dispersed material, and the moisture vapour transmission is ~5 g. of water per 100 sq.cm. per 24 hr. at 90°F. and at least 95% R.H.
S. V. S.

Mechanism of the Swelling and Dissolution of Xanthated Soda Cellulose Fibres—VIII (VI p. 467).

XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

Reactive Groups of Collagen. II—Reactions of the Carboxyl Groups. D. Burton, J. P. Danby, and R. L. Sykes. *J. Soc. Leather Trades Chem.*, **37**, 219–228 (July 1953).

Carboxyl groups in collagen are inactivated by esterification, using diazomethane, dimethyl sulphate, and methanol with acid catalysis, and by decarboxylation using Grignard reagents on methylated collagen, and lithium aluminium hydride on collagen. Measurements are made of methoxyl content, acid-binding capacity and *N*-alkyl content of esterified collagen and of acid-binding capacity, dicarboxylic acid content, and amide N content of the decarboxylated material. Esterification with diazomethane in neutral solution affects only a small percentage of COOH groups; there is considerable correlation between the number of unionised COOH groups and the extent of esterification. If the collagen is first brought into equilibrium with acid, there is increased, though still very slow, reaction. That the reagent is not specific is shown by a decrease in lysine ε-amino groups available to dinitrofluorobenzene. Dimethyl sulphate is better and causes little degradation; the ε-amino group of lysine is also alkylated. Methanol with acid catalysis is by contrast completely specific; it is shown that under anhydrous conditions the extent of esterification decreases as the size of the alcohol molecule is increased. Decarboxylation is efficiently carried out by means of lithium aluminium hydride, which also reduces some amide groups. The lower reactivity of Grignard reagents is partly due to steric hindrance. J. W. B.

Coloration of Leather. J. R. Blockey and D. H. Tuck. J.S.D.C., **69**, 273–279 (Aug. 1953).

"Crocking" or Loose Colour in Suède. C. T. Locke. *Leather Manfr.*, **63**, (11), **70**, (1952): *J. Soc. Leather Trades' Chem.*, **37**, 214 (June 1953).

It is suggested that buffing dust from the "pearl buffing" (i.e. the buffing before dyeing) plays a large part in this defect, and that if practicable a shaving should replace it. Thorough wetting of the crust for dyeing is important. Resin, or artificial rubber, emulsions in conjunction with the fat-liquoring are found useful: the following are suggested formulæ—

(*a*)		(*b*)	
2%	Sulphonated cod oil	4%	of 20% Casein solution
5%	Neoprene latex	4%	Suède oil
1%	Wetting agent	8%	Resin emulsion.
1%	Antioxidant		

As regards the type of dye to use, for blacks, diazotisation is to be preferred and for colours there is advantage in the metallised types, either the premetallised or those which are developed by afterchroming. In all cases reduction of concentration of dye in the dyebath is beneficial.
C. J. W. H.

Medullation in Black Horse-tail Hair. F. Kidd and C. E. Sagar. *J. Textile Inst.*, **44**, T 245 (May 1953).

Black horse-tail hairs reveal most commonly a lightly pigmented irregularly shaped central area surrounded by one which is much more heavily pigmented, and there has been controversy as to whether or not this is a medulla. It is found that ammoniacal picrocarmine solution, which stains cortical cells yellow and medullary cells red, behaves as expected with badger hair, kolinsky, black sable guard hairs, medullated white horse-tail hair, and medullated horse-mane hair, but that it stains red only parts of the central area of black horse-tail hair. This is, therefore, a medullated hair, though not over the entire lightly pigmented area. The medullation is unusual in that it is less well defined than in other hairs, and may be divided into several parts. J. W. B.

PATENT

Thermosetting Keratinoid Compositions. Rochester Button Co. *USP* 2,623,039

Keratinous substances, e.g. horn or feathers, are treated with a material which attacks the disulphide bonds, e.g. Na bisulphite or an alkylthiol, preferably in presence of a dispersing agent. They are then treated with phosphoric acid or a phosphate, and finally with a water-soluble calcium salt. The product hardens under heat and pressure into coherent translucent shapes of good strength and resistance to moisture and weathering.
C. O. C.

Fundamental Properties of Dyes (VIII p. 470).

XIII—RUBBER; RESINS; PLASTICS

Kinetics of the Hydroxymethylation of Phenols in Dilute Aqueous Solution. J. I. de Jong and J. de Jonge. *Rec. Trav. chim.*, **72**, 497–509 (June 1953).

Synthesis and Applicability of Block and Graft Copolymers. H. F. Mark. *Text. Research J.*, **23**, 294–298 (May 1953).

The terms *ordered* and *random normal copolymers*, and *block* and *graft copolymers*, are defined, and the properties to be expected from such polymers are discussed. The synthesis of graft copolymers by chain-transfer methods and by the use of reactive groups along the polymer chain is described, together with various examples of the methods of forming block copolymers. A. B.

Ion-exchange Resins in the Textile Industry. H. C. Borghetty, J. J. Bernard, and J. C. Winters. *Amer. Dyestuff Rep.*, **42**, P 338–P 342 (25 May 1953).

A survey of ion-exchange reactions includes a brief theoretical discussion and an account of applications to water softening, wool dyeing, the synthetic fibre industry, and automatic pH control in vat dyeing. J. W. B.

Depolymerisation Reactions in Vinyl Polymers. N. Grassie. *Chem. and Ind.*, 622–624 (27 June 1953).

A review of work in which the elucidation of the mechanism of the depolymerisation of polymethyl methacrylate, polystyrene, and polyethylene has been sought. Proposed methods for inhibiting this type of thermal degradation are mentioned. There are 12 references to the literature, 11 of which are readily accessible. J. W. D.

Pyrolysis of Vinyl Benzoate. R. J. P. Allan and P. D. Ritchie. *Chem. and Ind.*, 747 (18 July 1953).

Chitwood (*USP* 2,251,983) found that ethylene dibenzoate, a typical segment of the polyethylene terephthalate chain-molecule (I), yields, on pyrolysis at ~360–425°c., vinyl benzoate and benzoic acid in accordance with expectation. The thermal degradation of I has been found to yield COOH groups but not, however, vinyl ester groups. The authors, repeating Chitwood's work, find that CO and acetophenone are produced also. They advance evidence that the thermal breakdown occurs in two stages—

$$C_6H_5{\cdot}CO{\cdot}O{\cdot}CH{:}CH_2 \rightarrow C_6H_5{\cdot}CO{\cdot}CH_2{\cdot}CHO \quad (i)$$

$$C_6H_5{\cdot}CO{\cdot}CH_2{\cdot}CHO \rightarrow C_6H_5{\cdot}CO{\cdot}CH_3 + CO \quad (ii)$$

The intermediate β-ketoaldehyde is so unstable thermally, that it has so far been impossible to prove that (i) (similar to the Fries rearrangement) actually occurs; there is, however, experimental evidence for (ii). The discrepancy between the results of Chitwood and those obtained by the authors is explained in terms of "contact time" differences arising from the respective experimental conditions.
J. W. D.

PATENTS

Modified Polyvinyl Chloride Plastisol. Union Carbide & Carbon Corpn. *BP* 694,444

Modified plastisols of vinyl chloride polymers and copolymers, which, after heating to 160°c. in presence of a peroxide catalyst, give products of reduced flexibility, comprise a dispersion of the polymer (40–60%) in a mixture of conventional ester plasticiser (54–12%) and a polyethylene glycol dimethacrylate (6–28%) E. C.

Organo-tin Stabilisers for Polyvinyl Chloride. Advance Solvents & Chemical Corpn. *BP* 694,944

Non-volatile, non-hydrolysable organo-tin stabilisers for polyvinyl chloride, of formula $R^1{\cdot}O{\cdot}[SnR^2R^3{\cdot}O]_n{\cdot}R^1$ (R^1 = alkyl; R^2, R^3 = alkyl or aryl; $n > 1$) are prepared by reaction of $R^2R^3SnCl_2$ (1 mol.) with an alkali-metal alkoxide (< 1 mol.) or a mixture of an alcohol (< 1 mol.) with ammonia or an amine. E. C.

Propyl Dixylyl Phosphate. Celanese Corpn. of America. *BP* 695,762

Propyl dixylyl phosphate is prepared by heating to 270° C. isomeric xylenols (2 mol.) with phosphorus oxychloride (1 mol.) and reaction of the product with excess propanol at 45–75° C. The final product is suitable for use in lubricants and as a plasticiser for polyvinyl chloride, cellulose ethers and esters, and butadiene–acrylonitrile copolymers. E. C.

Hydrous Tribasic Lead Sulphate—Stabiliser for Vinyl Compositions. National Lead Co. *USP* 2,625,533

Hydrous tribasic lead sulphate $3PbO.PbSO_4.H_2O$ (production described in *USP* 2,249,330) is an excellent stabiliser for vinyl compositions when it is essential that the final product shall be transparent. C. O. C.

Crystalline Copolymers of 2:3-Dichlorobuta-1:3-diene. Firestone Tire & Rubber Co. *BP* 695,112

Polymers containing > 90% of 2:3-dichlorobuta-1:3-diene and having an intrinsic viscosity of 0·1–1·5 can be converted to oriented crystalline products of high tensile strength and flexibility. The intrinsic viscosity may be controlled within the desired range by several methods, e.g. by subjecting an infusible copolymer of dichlorobutadiene to a restrained chlorination confined to the introduction of 3–5% of chlorine based on the weight of the polymer. W. G. C.

Poly-imino Compounds. M. Erlenbach and A. Sieglitz. *BP* 696,364

Compounds which may be viscous liquids, soft rubber-like masses, or horn-like plastics are obtained by polymerising substances of general formulæ—

$$X{\cdot}Y^1{\cdot}N\begin{matrix}\diagup CR^1R^2\\ |\\ \diagdown CHR^3\end{matrix} \quad \text{or} \quad Y^2\left(O{\cdot}CO{\cdot}Y^1{\cdot}N\begin{matrix}\diagup CR^1R^2\\ |\\ \diagdown CHR^3\end{matrix}\right)_n$$

(Y^1 = aliphatic radical of > 4 C; Y^2 = aliphatic radical having O or S in the carbon chain; X = carboxylic salt, ester or amide or a nitrile group; R^1, R^2, and R^3 = H or Alk of > 4 C; n = 2–4), e.g. methyl β-ethyleneimino-propionate or 1:3-butylene bis-β-ethyleneimino-propionate. The products are useful as adhesives, surface coatings, or moulding compounds. W. G. C.

Colouring Amine–Aldehyde Resin Moulding Composition. Libbey-Owens-Ford Glass Co. *USP* 2,623,028

Much saving in time is obtained when preparing an amine–aldehyde moulding composition from "popcorn" (i.e. a dried filler impregnated with a thermosetting amine–aldehyde precondensate) by grinding the popcorn to powder, blending the pigment with some of this powder, and then blending this mixture with the rest of the ground popcorn. This method also has the advantage that the colour of the final product is under more accurate control. C. O. C.

Silicone-modified Alkyd Resins. Libby-Owens-Ford Glass Co. *BP* 694,716

Alkyd or oil-modified alkyd resins are heated with the product obtained by hydrolysing a mixture of a mono- or a di-alkyl-silicon chloride and a *cyclo*alkyl silicon trichloride to give resins which on curing at 150° C. show improved resistance to water, alkali, and weather. E. C.

Plastic Sheets having a Veined or Swirl-grain Pattern. Armstrong Cork Co. *BP* 697,110

Calendered jaspé sheets of the plastic material are cut into small strips, which are fed haphazardly to the rolls of a sheeting or blanket calender. The partly drawn mottling colour particles of the jaspé sheets form striated lines on the edge surfaces of the cut strips, and these lines form an excellent variegated marble graining in the final product. C. O. C.

Addition Products of an Amine and Sulphone-activated Ethylenic Compounds (III p. 455).
Polyesters, Polyamides, or Polyesteramides of High Molecular Weight (VI p. 469).
Methacrylonitrile Copolymers (VI p. 469).
Thermosetting Keratinoid Compositions (XII p. 476).

XIV—ANALYSIS; TESTING; APPARATUS

New Reagent for the Titration of Water. R. Belcher and T. S. West. *J.C.S.*, 1772–1776 (June 1953).

This new reagent for the determination of water in inert organic solvents consists of a solution of sulphur dioxide and bromine in chloroform, and is used similarly to the conventional Karl Fischer reagent, which is a solution of iodine, sulphur dioxide, and pyridine in methanol. Dioxan, benzene, pyridine, acetonitrile, and glacial acetic acid are less satisfactory than chloroform as solvent. The results are more nearly stoichiometric than those given by the Fischer reagent, whose drawback is also instability. The main disadvantage, however, is that alcohols interfere, but in most cases where a preliminary extraction of moisture is necessary, alcohol can be replaced by dioxan. H. H. H.

New Colorimetric Reagent for Carbohydrates. E. Lunt and D. Sutcliffe. *Biochem. J.*, **55**, 122–126 (Aug. 1953).

The use of a new colorimetric reagent, resorcinol-4:6-disulphonic acid, for the determination of hexoses and their polysaccharides is described; the reagent has been used chiefly for the determination of glucose, fructose, and their polysaccharides. From 10 to 250 μg. of glucose in 5 ml. may be determined with errors of < 1%. Inulin and starch give 100% and dextran 95% of the colour intensity given by their constituent monosaccharides. P. G. M.

New Techniques in Warp-size Preparation. A.A.T.C.C. Southeastern Section. *Amer. Dyestuff Rep.*, **42**, P 300–P 311 (11 May 1953).

Changes in the viscosity of warp sizes, from several types of starch, are followed during cooking by means of different viscometers; paste stability and the effect of high-pressure homogenisation are also studied. It is considered that there is scope for such instrumentation in ensuring that sizes are prepared within predetermined viscosity limits. J. W. B.

Effect of Wetting Agents on Adsorption of Crystal Violet by Glass. R. S. Subrahmanya, M. R. A. Rao, and K. S. G. Doss. *Proc. Indian Acad. Sci.*, **34 A**, 324–328 (1951): *Chem. Abs.*, **47**, 6734 (25 July 1953).

A study of the effect of Igepon T and Aerosol OT on the adsorption of Crystal Violet from aqueous solution by 100–150-mesh glass powder over the range pH 3·7–9·1. In absence of wetting agent adsorption rises from 0·013 mg. of dye per gram of glass powder at pH 3·7 to 0·089 mg. at pH 9·1. In acid solution dye adsorption rises steeply with small additions of wetting agent, and then decreases slowly to zero at high concentrations. Increase in pH lessens dye adsorption in presence of wetting agents. The method is suggested for comparing detergent action of wetting agents. C. O. C.

Single-number Systems for the Colour of Transparent Objects. P. Thomson. *J. Amer. Oil Chem. Soc.*, **30**, 259–262 (June 1953).

The author discusses the different systems of defining the colour of transparent objects, and describes a single-number system for the colour of transparent objects (cottonseed and soyabean oils) using a photoelectric instrument. The density or absorbence values of the oils are measured using blue and green light; red light may also be used in some cases. Equations have been formulated and are given for unbleached and bleached oils, incorporating these density values at the different wavelengths. From these equations the colours of unbleached and bleached oils may be expressed by a single number. P. G. M.

Determining Phenols and Tanning Agents. K. Tauböck. *Mitt. Versuchsanstalt Gärungsgewerbe und Insts. angew. Mikrobiol. Hochschule Bodenkult.*, **3**, 142–143 (1949): *Chem. Abs.*, **47**, 6310 (10 July 1953).

Xanthhydrol in strong acetic acid with H_3BO_3 as catalyst is a colour reagent for the microchemical detection of hydroxybenzenes, tanning agents, and phloricidin.

These compounds can be determined in very dilute extracts or small pieces of material. The sensitivity of the reaction increases with the number of OH groups in the compound to be determined. The lower limit of sensitivity of the reaction lies at 3–5 μg. Xanthhydrol is made by dissolving xanthene (25 g.) in NaOH (100 g.) in ethyl alcohol (1 litre), adding Zn dust (100 g.), and refluxing for 4 hr. The zinc hydroxide is filtered off, and the filtrate poured into water (5 litres). The xanthhydrol is filtered off, washed with water, and dried *in vacuo*. Xanthhydrol gives no reaction with flavones, flavanols, or anthocyanins. C. O. C.

Identification of Amines by X-Ray Powder Patterns of their Chloroplatinates. C. W. Gould and S. T. Gross. *Anal. Chem.*, **25**, 749–751 (May 1953).

Submilligram quantities of volatile amines can be characterised by conversion into their chloroplatinates, which can be identified by means of their X-ray diffraction patterns; the method avoids the micromanipulations necessary for the preparation and purification of other derivatives. Diffraction data for 25 common aliphatic and aromatic amines are given. J. W. D.

Oxidation of *N*-Alkylarylamines—Identification of *N*-Alkyl Groups. F. W. Neumann and C. W. Gould. *Anal. Chem.*, **25**, 751–759 (May 1953).

The scope and the limitations of the oxidation of *N*-alkylarylamines by dichromate in a sulphate–bisulphite buffer as a method for the characterisation of *N*-alkyl groups are studied; simple methods are considered for the identification of the products of the degradation. Simple mg.-scale techniques are detailed, and there are diagrams of the apparatus. Of the 76 aromatic amines examined, the majority yielded aromatic aldehydes in addition to quinones and coloured condensation products on oxidation. Compounds containing *para* groups which promote ring oxidation, e.g. OH,NH_2, and NH·CO·R gave, instead of aldehydes, the corresponding aliphatic amines. Both types of oxidation were demonstrated with certain aminophenols and substituted *p*-phenylenediamines. Simple aliphatic amines are not oxidised under the conditions used. Tables show the amines which were examined and indicate the mode of their degradation, whilst others list numerous physical constants of the derivatives of many of the oxidation products. Alcohols interfere, since they, too, are oxidised to aldehydes; it is possible also that certain functional groups, not yet studied, may interfere similarly. A few amines fail to react because of extreme insolubility in the chromic acid reagent. There is extensive discussion of the reaction conditions. Fifty-five references to the literature are provided. J. W. D.

Substituted Benzidines and Related Compounds as Reagents in Analytical Chemistry. XII—Reagents for the Precipitation of Sulphate. R. Belcher, A. J. Nutten, and W. I. Stephen. *J.C.S.*, 1334–1337 (May 1953).

The sulphates of the 4-amino-4′-halogenodiphenyls are described, and their solubilities in water found to be considerably less than those of 4-amino- and 4:4′-diaminodiphenyl. In particular, the least soluble sulphate is that of 4-amino-4′-chlorodiphenyl, and this is developed as a new reagent for the rapid and accurate alkalimetric determination of the sulphate ion. H. H. H.

Methylation as an Aid in the Analysis of Polynuclear Aromatic Compounds. R. L. Cooper. *Chem. and Ind.*, 516 (23 May 1953).

Micro-quantities of polynuclear hydrocarbons in complex materials can be detected and determined by a combination of chromatography and absorption spectrophotometry. Quinones and anthrones have absorption spectra which are too poor to be of use, but the corresponding methoxy derivatives are much better and, moreover, in many cases yield information regarding the position and the number of oxygen atoms in the original compound. Quinones may be separated prior to methylation, or by virtue of the increase in the degree of adsorption on alumina resulting from an increase in the number of $O{\cdot}CH_3$ groups in the molecule. Hydroxy derivatives are difficult to separate, are often unstable on alumina, and should first be methylated. The technique is applicable with advantage to sulphonic acids and carbazoles. Azahydrocarbons do not require methylation (which would in any case be inapplicable) and may also be separated upon alkaline columns. Mixtures containing a few μg. each of anthracene, anthrone, and anthraquinone have been subjected to reductive methylation, and the products separated, identified, and determined. J. W. D.

Polarographic Determination of Anthraquinone. R. L. Edsberg, D. Eichlin, and J. J. Garis. *Anal. Chem.*, **25**, 798–800 (May 1953).

Anthraquinone may be determined polarographically in the presence of anthracene, phenanthrene, carbazole, maleic anhydride, and phthalic anhydride. Polarograms are obtained in soln. of *NN*-dimethylformamide 0·1 N, in LiCl, containing 0·2 ml. of 0·5 M aq. LiOH to eliminate anhydride interferences. Anthraquinone exhibits waves at $E_{\frac{1}{2}} = -0{\cdot}83$ v. and $E_{\frac{1}{2}} = -1{\cdot}17$ v. against the saturated calomel electrode, representing a reduction through the semiquinone to anthraquinol. Though the calculated electron changes are 0·72 and 0·79 (rather low for a clear-cut one-electron-change reaction), supporting evidence shows it to be clearly a reversible reaction involving two one-electron-change reactions. The apparatus and the procedure are detailed. J. W. D.

Determination of Ferulic Acid in Curcumin. T. Kimura. *Ann. Rept. Takeda Research Lab.*, **11**, 59–61 (1952): *Chem. Abs.*, **47**, 6312 (10 July 1953).

The red coloration of ferulic acid with *p*-$ClN{:}N{\cdot}C_6H_4{\cdot}SO_3H$ is directly proportional to the amount of the acid present; the acid is estimated by measuring the extinction coefficient of the coloured soln. in a Beckmann spectrophotometer at 4700A. C. O. C.

Analysis of Flavones. I—Stoichiometry of Dissolved Metal-salt Complexes of Rutin and Quercetin. L. Hörhammer and R. Hänsel. *Arch. Pharm.*, **285**, 438–444 (1952); *Chem. Abs.*, **47**, 8322 (25 Aug. 1953).

The stoichiometric composition of the complexes of rutin and quercetin with Zr (IV), Al (III), and Cu (II) was examined photometrically. All complexes have a 1 : 1 flavone : metal ratio except the quercetin–Al complex, which consists of 1 Al : 2 quercetin. The Zr complexes, unlike the others, are stable in presence of strong acids (2 M $HClO_4$). Quercetin can be differentiated from its 3-glucosides by the difference in stability of the Zr complex in presence of citric acid; the aglucone appears to have a greater affinity for Zr. C. O. C.

Determination of the Pigments of Red Wine. V. V. Williams and R. D. Taranova. *Vinodelie i Vinogradarstvo S.S.S.R.*, **10**, (4), 29–30 (1950): *Chem. Zentr.*, **1**, 536 (1951): *Chem. Abs.*, **47**, 8310 (25 Aug. 1953).

The method described depends upon measurement of the extinction at wavelength 530 mμ. with the Pulfrich step photometer. Oenin and œnidin, isolated by a modified picrate method, were used to obtain a calibration curve. The extinction of the sum of the œnin and œnidin varied only slightly from the extinction of the wine. C. O. C.

Particle-size Measurements on Paints. W. Garmsen. *Farbe und Lack*, **59**, 92–97 (1953): *Chem. Abs.*, **47**, 6669 (10 July 1953).

Because of the unfavourable effect on paints of a very minute proportion of coarse particles, it is more important to know the size of the coarsest pigment particles than the size distribution of the remainder. This information is easily obtained by use of the Hegman grind gauge (cf. Fasig, *Drugs, Oils and Paints*, **54**, 438 (1938)) or its German counterparts (Goeb, *Chem. Abs.*, **45**, 3165 (1951); Arnold, *ibid.*, **46**, 6399 (1952)), but this device has many sources of error (Doubleday and Backman, *Amer. Paint J.*, **34**, (38), 72–80; (39) 78–90 (1950)); the major inaccuracy derives from the fact that the paint does not fill the channel completely. A device designed to overcome the deficiencies of the Hegman gauge consists of two mirror glass plates, 122 × 48 mm., 12 mm. thick, with a centimetre scale etched into their edges, and a selection of exchangeable gauges (60, 80, 100, 150, and 300 μ. thick). The selected gauge is placed on one of the plates at the 10 cm. mark, the paint is poured on, the second plate is placed above the first, and the two plates are held together by a strong clamp at each end; after removal of excess paint followed by a short interval, the clamps are removed, and the plates carefully slid apart along their contact level. In this way each plate retains half of the paint wedge (as is confirmed by the wet gauge). Immediately (15 sec. for cellulose nitrate lacquers, 1 min. for paints or enamels) after separation, the surface is scanned for the easily countable

visible particles, and their number noted along with their position on the cm. scale. Each cm. equals 5% of the gauge thickness. From these data and the gauge thickness, the prevailing particle size (location of counting limit) and the distribution of the coarser particles are determined. The results are reproducible regardless of the size of gauge used. The applications of the device are discussed.
C. O. C.

Detection of Aluminium Compounds in White Pigments. E. F. M. Waterman-Schrijver. *Verfkroniek*, **26**, 57 (1953): *Chem. Abs.*, **47**, 8387 (25 Aug. 1953).

Detection of Al in white or near-white pigment mixtures (cf. Fergl, *Qualitative Analysis by Spot Tests* (1939), p. 116) is not specific in presence of Ti, which gives the same red coloration. Therefore, Ti must be removed from the HCl filtrate before the Al reaction by making the HCl soln. alkaline with KOH and filtering it after boiling. This filtrate is then made faintly acid and used for the Al test.
C. O. C.

Single-fibre Load Elongation Tester for Cotton. R. S. Orr and J. N. Grant. *Text. Research J.*, **23**, 505–509 (July 1953).

An apparatus is described for the production of load–extension curves in addition to end-point data on cotton fibres. Devices are incorporated permitting cyclic loading studies and static life determinations.
A. B.

Shearing of Filaments and Yarns. S. Backer. *Text. Research J.*, **23**, 438–440 (June 1953).

A method is described for investigating the shear strength of fibres, and this letter reports microscopic observations of the cutting action which occurs in shearing tests.
A. B.

Qualitative Application of Iodine Absorption in the Testing of Fibrous Materials. K. Schwertassek. *Faserforsch. und Textiltech.*, **4**, 184–187 (May 1953).

A discussion is given of the use of qualitative iodine absorption tests, in which observations are made on the colour acquired by the fibre when it absorbs iodine from dil. soln. (saturated with Glauber's salt), or is treated first in a conc. iodine soln. and then in running water to bring about desorption. Normal native and regenerated celluloses become yellow or brown in the adsorption test, and blue and violet respectively in the desorption test. Amorphous cellulose formed from the normal fibres by crushing gives, however, a violet colour in the absorption test, and it is noted that the preparations so obtained become cemented together when moistened and then dried (recrystallisation); the significance of this effect in papermaking is discussed. Local damage in fabrics produced by acids and oxidising agents is indicated in the tests by light places, but, in the absorption test, caustic alkali splashes on viscose rayon fabric show as darker places (in the desorption test, 6% NaOH gave lighter, but 8% and 10% NaOH darker, places). The use of the method in the detection of uneven kiering is discussed.
A. E. S.

Conditioning of Cotton, Linen, Hemp, Wool, and Rayon Staple Fibres. SNV 9 64 41. Schweizerische Normen-Vereinigung. *Textil-Rund.*, **8**, 313–318 (June 1953).

These directions solely define the technical conditions for testing, leaving decisions regarding the number and choice of bales to be sampled, method of sampling, regains, tolerances, compensation, etc. to regulation by technical associations or agreement between buyer and seller.
H. E. N.

Application of Identification Tints, with particular reference to Acetate Rayon Staple. H. C. Olpin and A. J. Wesson. J.S.D.C., **69**, 357–362 (Oct. 1953).

Allwörden Reaction. A. Parisot and M. Leveau: E. H. Mercer and R. L. Golden. *Text. Research J.*, **23**, 440–441 (June 1953).

A previous article on this subject (E. H. Mercer and R. L. Golden, *Text. Research J.*, **23**, 43 (1953)) is discussed, and it is considered that the whole cuticle is involved and not the epicuticle.
A. B.

Quantitative Determination of Ardil when mixed with Wool. R. Preston and N. Warburton. *J. Textile Inst.*, **44**, T 298–T 305 (June 1953).

Methods are investigated for determining Ardil in admixture with wool, and the following procedure is considered satisfactory—A 1-g. sample of degreased air-dry blend is added to 100 ml. of a boiling solution containing 5 g. of barium hydroxide, and 20 g. of sodium or potassium nitrate, and boiled for 6 min. The mixture is filtered hot through a sintered glass crucible (Pyrex S.F. 2B1), and the residue washed three times with 20 ml. of hot 2 N-HCl, then with hot water, and finally with acetone, and dried at 95–100° C. Several examples are provided of cloth and yarn analyses.
J. W. B.

Detection of Incompletely Drawn Perlon and Nylon Filaments. W. Pelz and F. Harbeck. *Faserforsch. und Textiltech.*, **4**, 304–306 (July 1953).

A Perlon material is immersed in a cold 3% soln. of Rhodamine, in which incompletely drawn filaments, and also foreign matter such as size, become deeply stained. The material is then treated in chromic acid (CrO_3 (10 g.), H_2SO_4 (11 ml.), and water (to 100 ml.)), when effects due to foreign matter disappear, and is examined under the microscope. Fully drawn filaments have a roughened appearance, whereas undrawn or incompletely drawn filaments are more heavily stained and are clear and structureless in appearance.
A. E. S.

Rapid Test for Polyacrylonitrile Fibres. M. Sorkin. *Textil-Rund.*, **8**, 321 (June 1953).

To the fibres in a little water is added a solution prepared from 8 c.c. saturated copper sulphate soln. and 0·2 g. diphenylamine in 400 c.c. sulphuric acid. Sulphuric acid liberates hydrocyanic acid from polyacrylonitrile, and this converts copper sulphate into cuprous cyanide and an oxidising agent, which yields an intense blue colour with diphenylamine. Other oxidising agents must be absent.
H. E. N.

Analysis of Cotton–Glass–Asbestos Yarn. C. L. Hilton. *Text. Research J.*, **23**, 401–404 (June 1953).

A method of analysis is given for the determination of cotton–glass–asbestos yarns, based on carbon, hydrogen, and ash determinations after removal of finishing agents from the sample.
A. B.

Determination of Resistance to Washing of Cotton and Linen Textiles. SNV 9 87 41. Schweizerische Normen-Vereinigung. *Textil-Rund.*, **8**, 371 (July 1953).

Detailed method.
H. E. N.

Standard Method for Determination of Shrinkage on Washing of Woven Rayon and Synthetic-fibre Fabrics. Tentative Textile Specification No. 24, 1953. *J. Textile Inst.*, **44**, S 1–S 4 (July 1953).

Laboratory Investigation of Methods for Determining Shrinkage on Washing of Woven Rayon and Synthetic-fibre Fabrics. *J. Textile Inst.*, **44**, S 5–S 41 (July 1953).

A detailed account is presented of work carried out in formulating a shrinkage test for a textile specification.
J. W. B.

Measurement of Redox Potentials. K. Edelmann. *Faserforsch. und Textiltech.*, **4**, 337–342 (Aug. 1953).

A discussion of the significance of redox potentials, their measurement, and types of apparatus used.
A. E. S.

Observations on some Commercial Colour-matching Lamps. J. C. Guthrie and P. H. Oliver. J.S.D.C., **69**, 430–435 (Nov. 1953).

Matching U.S.A.F. Silvertan Shade No. 193. H. R. Davidson and R. D. Haire. *Amer. Dyestuff Rep.*, **42**, 253–257 (27 April 1953).

In the award of dyeing contracts the tolerance limits between the standard and the submitted samples are seldom specified, leading to submission of both useless and unnecessarily precise samples. The current practice in the United States Air Force in accepting samples for Silvertan Shade No. 193 (which has given much trouble) on wool gabardine is studied, 66 samples, 30 of which were accepted, being measured spectrophotometrically. Calculations are made with the automatic tristimulus integrator; tristimulus data and Munsell notations are tabulated, and the results illustrated graphically, including % reflectance–wavelength curves. It is shown that, if a sample lies well within an ellipse about 1·7 times as large as the MacAdam sensitivity ellipse, if it lies well within an ellipse about 2·5 times as large as the visual sensitivity ellipse when viewed under tungsten light, if it does not differ from the standard in Y value by $>$ ca. $\pm$ 0·02 and if its spectrophotometric curve is nearly identical in shape with that of the standard, then

it will be accepted as a colour match. The method is applicable to any colour, and much time could be saved by providing manufacturers with the appropriate spectrophotometric requirements. J. W. B.

Uses of Microscopy in Textile Dyeing and Finishing. G. G. Taylor and J. C. Brown. J.S.D.C., **69**, 396–403 (Nov. 1953).

International Fastness Tests. International Standards Organisation. J.S.D.C., **69**, 409–429 (Nov. 1953).

Development of the Geometric Grey Scales for Fastness Assessment. Society of Dyers and Colourists Fastness Tests Co-ordinating Committee. J.S.D.C., **69**, 401–409 (Nov. 1953).

Development of the Geometric Staining Scale. K. McLaren. J.S.D.C., **69**, 285–287 (Aug. 1953).

Anomalous Fading in the Fadeometer. A.A.T.C.C. Research Subcommittee on Fastness to Light. *Amer. Dyestuff Rep.*, **42**, P 379–P 385 (8 June 1953).

Increasing use of new fibres, and of vat dyes on wool, has disclosed more instances of anomaly between fading-lamp and sunlight fastness ratings. Accordingly, a large number of test specimens are exposed to sunlight under different conditions of screening, and from the results a number are selected for comparative fading-lamp trials. A preliminary description of the results is given. J. W. B.

Colorimetric Method for the Preparation of Colour-blindness Tests. E. N. Yustova. *Doklady Akad. Nauk S.S.S.R.*, **90**, 533–535 (1 June 1953).

Colour-blindness tests of the Ishihara type are constructed from diagrams obtained by the use of a trichromatic colorimeter. These diagrams are projections on the *rg* and *rb* planes of three-dimensional plots expressed in the co-ordinates of the colorimeter (*r*, *g*, and *b*), and they consist of curves, each of which represents a given hue and is graduated in units of lightness; these curves radiate from the point representing the standard light source. Projections of the axes of the fundamental physiological co-ordinates (*R*, *G*, and *B*), as established by this school of workers (Rautian, Yustova, *et al.*), are also represented on the diagrams. When points representing two particular colours fall on a line parallel to the *G* axis in *both* diagrams, then these colours will be indistinguishable to a green-blind person. The treatment of red-blindness is analogous. Owing to the low relative brightness of the *B* co-ordinate, a simplified treatment, requiring one diagram only, is possible for blue-blindness. A. E. S.

Radioactive Isotopes in Roller Printing. W. Meitner and H. B. Hampson. J.S.D.C., **69**, 283–284 (Aug. 1953).

Identification of Textile Finishes. R. Krammes and C. Maresh. *Amer. Dyestuff Rep.*, **42**, 317–327 (25 May 1953).

A comprehensive account of methods for examining textile finishes, based on their removal by different solvents microscopic examination, staining tests, and specific tests for identifying the product when it has been removed from the fibre. J. W. B.

Proposed Laboratory Accelerated Ageing Test for Chlorine Retention in Textile Fabrics. G. S. Wham and P. B. Mack. *Amer. Dyestuff Rep.*, **42**, 455–456 (20 July 1953).

A laboratory test for the chlorine retention of resin-treated rayon fabric comprises placing the material in distilled water containing 0·13% available Cl (from NaOCl), raising the temperature to 140°F., and stirring for 10 min. The fabric is then rinsed thoroughly five times, for 30 sec. each, in distilled water with vigorous stirring, and dried. Specimens are exposed for 40 hr. in a standard Fadeometer, removed, and pressed with a hand iron for 80 sec. at 275°F., the iron being lifted after each 10 sec. of contact. Strength tests are finally made. Results are shown to compare favourably with extensive practical launderings trials. J. W. B.

PATENTS

Determining the Condition of Water as regards its Content of Predetermined Types of Impurities. Filtrators. *BP* 695,623

Automatic apparatus for determining at fixed intervals of time the amounts of one or more impurities in water. C. O. C.

Measuring, Recording, or Controlling pH. George Kent. *BP* 695,909–11

Pulsed apparatus is described which is unaffected by variations in the temperature of the liquid whose pH is being measured. C. O. C.

Inspecting the Fluorescent Properties of Materials. Metropolitan-Vickers Electrical Co. *BP* 696,098

Apparatus independent of artificial sources of ultraviolet radiation comprises a container in which may be placed a tray containing the sample. The container is made of a material which is transparent to ultraviolet radiation but opaque to light and has an eyepiece through which the sample can be viewed. C. O. C.

3:4-, 3:5-, and 3:6- Dimethylcatechol (IV p. 456).

Thermal Decomposition of Hydrazobenzene, studied by the Use of Nitrogen Isotopes (IV p. 457).

Chromatography of Synthetic Dyes (IV p. 458).

Origin and Development of Titanium Dioxide Pigments (IV p. 462).

Investigation of the Frictional Properties of Textile Fibres under Variable Fibre Stress (VI p. 465).

Mechanism of the Formation of Electrostatic Charges on Fibres (VI p. 466).

Chlorine Retention in Textile Finishes (X p. 472).

Preliminary Steps in the Development of an Accelerated Laboratory Test for Chlorine-retention Properties in Resin-treated Fibres (X p. 472).

Preliminary Steps in the Development of an Accelerated Laboratory Test for Chlorine-retention Properties in Resin-treated Fabrics (X p. 472).

Further Steps in the Development of an Accelerated Laboratory Test for Chlorine-retention Properties in Resin-treated Fabrics (X p. 472).

Manufacture, Structural Design, and Testing of Felts for the Papermaking and Allied Industries (XI p. 474).

Fractionation as a Method of Studying the Uniformity of Carboxymethyl Celluloses (XI p. 474).

XV— MISCELLANEOUS

New Nuclear Staining Method with a Vegetable Indicator Dye, Sambucyanin, from *Sambucus nigra*. A. Novelli. *Experientia*, **9**, 152–153 (1953): *Chem. Abs.*, **47**, 7011 (25 July 1953).

The dye obtained by extracting elderberries with 30% ethyl alcohol varies in colour from red at pH 1·0–2·5 to deep blue at pH 10. As a microscopic stain it reacts selectively with cell nuclei, giving sharpness of detail without tending to overstain. C. O. C.

Factors influencing Control of Colour in Amber Glasses. R. W. Hopkins and W. H. Manring. *Glass Ind.*, **34**, 251–254, 276 (1953): *Chem. Abs.*, **47**, 7177 (25 July 1953).

Control of colour centres around retention of sulphur and formation of sulphide groupings are discussed. Stability of colour and the hue are associated with the amount and valency of the iron present. Low iron content gives reddish, excess of reduced iron gives brownish, while excess of oxidised iron results in greenish tints. To produce stable commercial amber glass of transmission 30–50% at 550 mμ., a combination of Fe and Mn oxides must be present in excess of the amount needed for the sulphide grouping. C. O. C.

Metallising of Glass, Ceramics, and Plastic Surfaces. R. J. Heritage and J. R. Balmer. *Metallurgia*, **47**, 171–174 (1953): *Chem. Abs.*, **47**, 6849 (25 July 1953).

The three important techniques for coating with metal— reduction from aq. soln., reduction by heat, and evaporation *in vacuo*— are discussed in detail. C. O. C.

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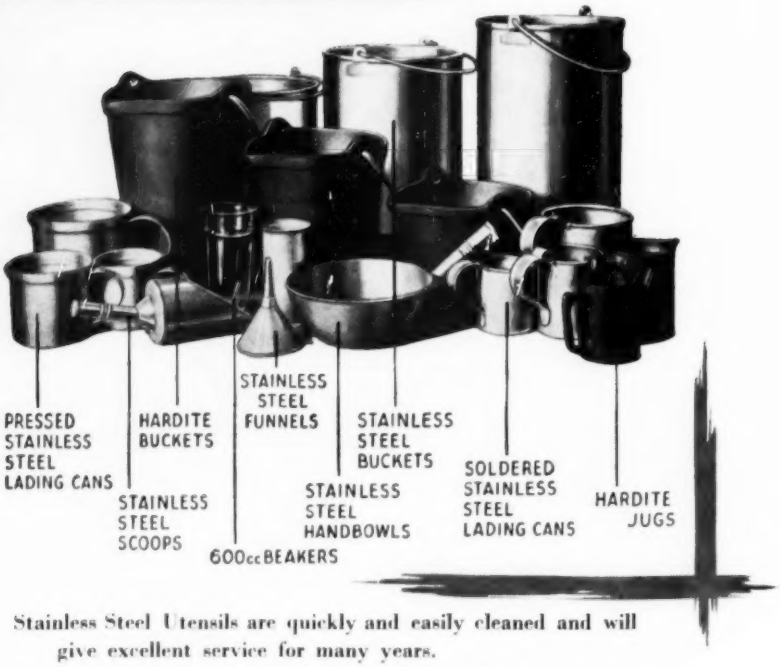

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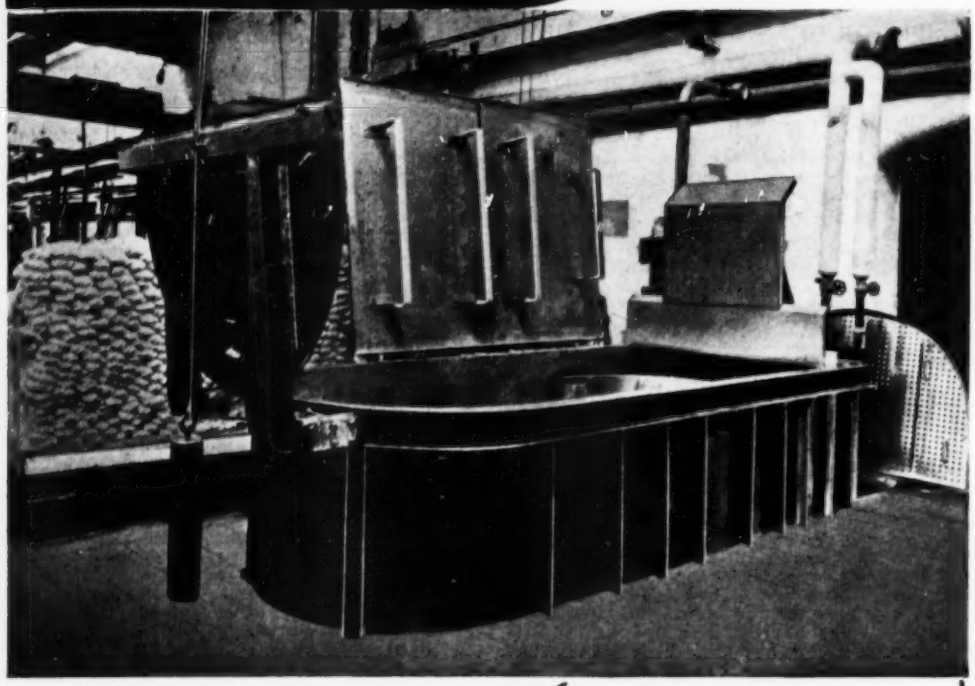

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FORTHCOMING MEETINGS OF THE SOCIETY — *continued from page x*

MANCHESTER JUNIOR BRANCH

All lectures to be held in the Reynold's Hall, Manchester College of Technology, at 6.30 p.m.

1953	
Wednesday 16th Dec.	J. S. Ward, Esq., B.Sc. *Recent Developments in the Dyeing of Synthetic Fibres*
1954	
Monday 1st Feb.	C. L. Bird, Esq. *Theoretical Aspects of the Dyeing of Cellulose Acetate Rayon*
Monday 8th March	Lecture, title to be announced later

HUDDERSFIELD SECTION

All lectures at Field's Cafe, Westgate, Huddersfield, at 7.30 p.m. unless otherwise stated

1953	
Monday 23rd Nov.	J. L. Beadle, Esq. (Brown & Forth Ltd.). *Orlon Acrylic Fibre* Joint with Huddersfield Textile Society, to be held in Large Hall, Huddersfield Technical College

HUDDERSFIELD SECTION (*continued*)

Tuesday 8th Dec.	Dr. Baines. *Photography as a Scientific Implement* Joint with Huddersfield Section of Royal Institute of Chemistry
Tuesday 15th Dec.	I. D. Rattee, Esq. (I.C.I. Ltd.). *Level Dyeing of Wool with Afterchrome Dyes*
1954	
Tuesday 19th Jan.	F. Thies, Esq. (Messrs. B. Thies, Coesfeld). *High Temperature Dyeing and Pressure Drying*
Tuesday 16th Feb.	J. G. Grundy, Esq. (Clayton Aniline Co. Ltd.). *Cibalan Dyes and their Fastness Properties on Nylon and Application to Wool-Nylon Unions*
Tuesday 16th Mar.	ANNUAL GENERAL MEETING, followed by a lecture, *Fastness Properties for Users Satisfaction.* J. S. Ingham, Esq. (Marks & Spencer Ltd., London)

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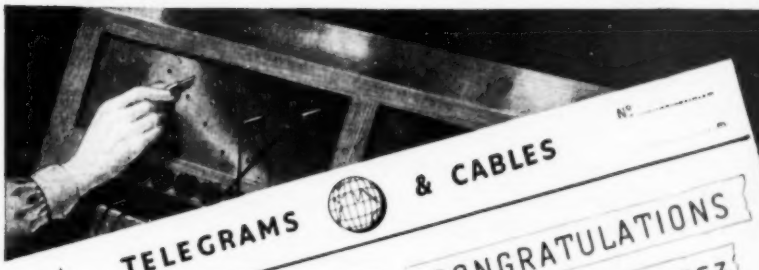

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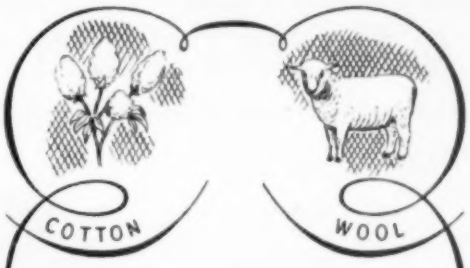

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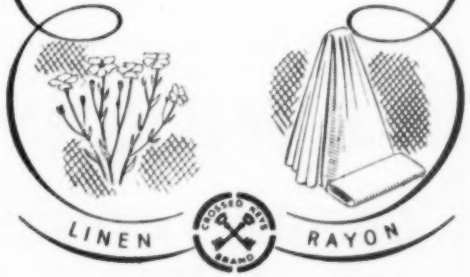

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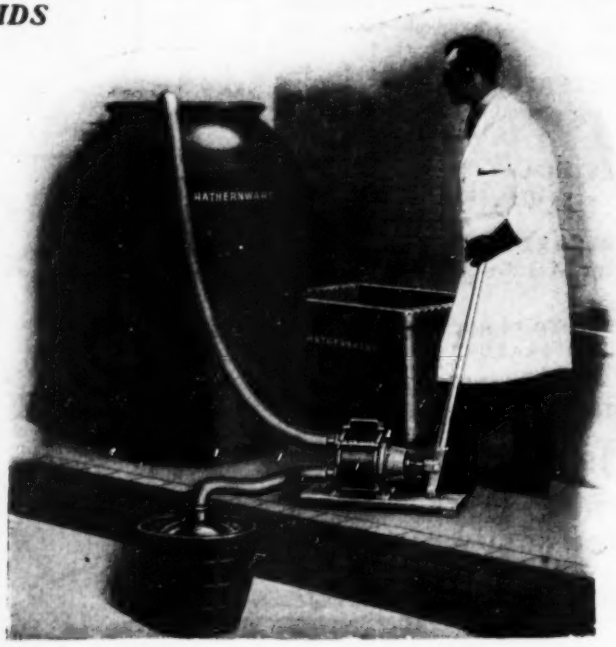

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